U.S. Mines, Bureau of

Bulletin 112

DEPARTMENT OF THE INTERIOR

FRANKLIN K. LANE, SECRETARY

BUREAU OF MINES

VAN. H. MANNING, DIRECTOR

MINING AND PREPARING DOMESTIC GRAPHITE FOR CRUCIBLE USE

BY

GEORGE D. DUB and FREDERICK G. MOSES

WITH A

CHAPTER ON METHODS OF ANALYSIS USED BY THE BUREAU OF MINES

BY

G. B. TAYLOR and W. A. SELVIG

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First edition. January, 1920.

CONTENTS.

TABLES.

ILLUSTRATIONS.

MINING AND PREPARING DOMESTIC GRAPHITE FOR CRUCIBLE USE.

By George D. Dub and Frederick G. Moses.

PREFATORY STATEMENT.

The Bureau of Mines, in connection with the investigations of war minerals it conducted, examined the graphite deposits of this country, studied the methods of mining and preparation used, and sought to devise more efficient methods for the preparation of domestic graphite, and to standardize the quality of the product. The graphite investigations covered three general phases, as follows:

1. An examination of the deposits in Alabama and other States, and a survey of the methods of mining and preparation used. In this connection methods of sampling and analysis were noted and experiments made to determine a standard method of sampling and a rapid but accurate method of analysis.
2. Experimental work on the concentration and refining of domestic crucible graphite to improve the quality of the product and to lessen waste.
3. Experimental work in crucible manufacture to determine the properties of domestic flake and the maximum proportions that might be used without impairing the quality of the crucibles. This work was accompanied by photomicrographic study of crucible structure.

In this bulletin are presented the results obtained in the first two phases of the work outlined above. The bulletin is in two parts. The first part describes the methods of mining and milling used, suggests a standard method of sampling finished graphite, and describes a rapid and convenient method of analysis developed at the Pittsburgh station of the Bureau of Mines, to which had been assigned the analytical and microscopic work. The second part describes experiments on the concentration and milling of graphite, which were made at the Salt Lake City, Utah, station. As soon as the reports incorporated in this bulletin were ready, mimeograph copies were distributed to those persons most interested in order to make the results immediately available.

The experimental work on crucible manufacture, assigned to the Columbus, Ohio, station, yielded results which were made known to the industry. A report on this work will be published later as a separate bulletin.

MINING AND MILLING METHODS IN ALABAMA.

By George D. Dub.

INTRODUCTION.

The writer, in connection with the graphite investigation of the Bureau of Mines, war minerals investigations, was charged with the examination of mining and milling methods. During the course of the field work, Alabama, New York, Pennsylvania, and Texas, the principal producing districts in the United States, were visited. In addition, the writer visited a plant in which graphite is refined from "kish," a by-product obtained from iron blast-furnaces and steel plants. The field work was planned to permit of studying the methods of mining and milling and of refining graphite for market. The results of this observation and study are set forth in the following pages. A standard method for sampling finished graphite is suggested, and a rapid, convenient method of analysis used by the bureau is described.

ACKNOWLEDGMENT.

Acknowledgment is made of the whole-hearted cooperation of producers in all of the fields. The Graphite-Producers Association of Ashland, Ala., the members of the staff of the office of the State geologist of New York, and of the various bureaus and boards in Washington have been of great assistance in furthering the aims of this investigation and in making effective various recommendations made from time to time. To H. S. Mudd, mineral technologist of the Bureau of Mines, special credit is due for his painstaking review of the manuscript.

LITERATURE.

A bibliography on graphite has been prepared by Ferguson [a]. The Canadian Bureau of Mines is revising Cirkel's monograph on graphite [b], Prof. B. L. Miller is revising Cirkel's monograph on deposits of Pennsylvania [c] for the State geological survey, and the

[a] Ferguson, H. G., Graphite in 1917: Mineral Resources, U. S., 1917, U. S. Geological Survey, 1918, pp. 117–119.

[b] Cirkel, Fritz, Graphite; its properties, occurrence, refining and uses: Canada Department of Mines, Mines Branch, 1907, 307 pp.

[c] Miller, B. L., Graphite deposits of Pennsylvania: Topographic and Geol. Survey of Pennsylvania, 1912, 147 pp.

New York State deposits are fully described in a bulletin of the State Museum by H. L. Alling[a]. In view of the detailed geological discussions and the full descriptions of milling and refining apparatus and of methods in the publications mentioned, such phases receive only incidental mention in this report. No statistics except those not available in State or United States Geological Survey bulletins have been incorporated.. The aim is simply to describe present practice and recent developments.

DEVELOPMENT OF GRAPHITE INDUSTRY IN THE UNITED STATES.

Before 1915 nearly all the graphite used in crucible manufacture in this country was imported. Little domestic graphite was used in crucibles, that mined being used chiefly for the manufacture of lubricants, paint, foundry facings, and other purposes. In 1915 the demand for graphite crucibles increased greatly, because of the placing of large foreign contracts for munitions and ordnance with American plants, and because of the inferiority of crucibles made from clay other than that from Klingenburg, Bavaria, which had been cut off by the blockade. Certain crucible manufacturers, fearing a shortage of foreign graphite, offered high prices for domestic flake graphite. This stimulated development of the graphite-bearing rocks of Alabama. Also, the uncertainty regarding foreign supplies caused crucible manufacturers to use a larger proportion of domestic graphite in their mixtures than they had ever before attempted.

During 1916 and 1917 the amount of graphite imported was about eight times the domestic production. Imported graphite comes mainly from Ceylon, Madagascar, and Korea. The amorphous graphite from Korea can be easily replaced by that from Mexico, or, if necessary, by development of somewhat lower grade deposits in the United States. Madagascar and Ceylon have furnished about 70 per cent of the total graphite imported, and of this amount approximately 90 per cent has been used in crucible manufacture.

Until the United States entered the war in 1917, the use of Ceylon and Madagascar graphites by American manufacturers was unrestricted as long as guaranties could be furnished to the British and French Governments that the products into which these graphites entered would not fall into the hands of the enemy. On the declaration of war in April, 1917, these guaranties became unnecessary, as they were superseded by broader American defense measures.

During the interval in the winter of 1917–18 when freight conditions were most congested, an embargo against the shipment of

[a] Alling, H. L., The Adirondack Graphite Deposits, N. Y. State Museum Bull. 199, 1917, 150 pp.

domestic graphite was ordered and permitted to remain in force until the early part of March, 1918. The result of this embargo was a stagnation of the domestic mining industry. The removal of the freight restriction on domestic graphite was followed in April by a complete embargo on importations of overseas graphite during the interval April 15, 1918, to July 1, 1918. For the remainder of the calendar year 5,000 long tons was to have been permitted to enter this country. This order was modified to the extent of allowing importations during the period April 15, 1918, to July 1, 1918; the total amount of these imports, however, was to have been deducted from the 5,000 tons scheduled to enter during the last six months of the year.

Toward the end of June, 1918, in view of the necessity for conserving shipping for the direct military program and after stocks of overseas graphite in the hands of crucible makers, refiners, and dealers were found sufficient to last about six months, it was decided after July 2, 1918, to restrict completely the importation of overseas graphite for the rest of 1918. This order was followed on August 10, 1918, by a request from the War Industries Board that all crucible makers use 20 per cent domestic flake graphite in their crucible graphite mixtures for the rest of 1918 with an increase of 25 per cent for 1919. This request carried with it the statement that applications for import licenses of manufacturers not complying with the provisions of the request would not be approved by the War Industries Board.

The War Trade Board ruling of July 2, 1918, was superseded on October 17, 1918, by a ruling permitting imports of overseas graphite by manufacturers whose applications had been approved by the War Industries Board. The purpose of this ruling was to permit the carrying of 3 or 4 months' supply of graphite, in view of the length of time required for shipments from Ceylon and Madagascar to reach the factory in which they were to be used.

ESTABLISHMENT OF PERMANENT DOMESTIC INDUSTRY.

Before the declaration of war in 1914, all crucible makers in the United States, without exception, used clay imported from Klingenburg, Bavaria, in the manufacture of crucibles. Little work of any kind had been done with domestic or English clay. With the declaration of war the cutting off of Bavarian clay forced the crucible maker to turn his attentions to other clays. Although at first serious difficulties were encountered, the clay problem is fairly well in hand with the result that crucible efficiency compares favorably in service with prewar standards. After clay difficulties had been largely surmounted, the problems of crucible manufacture were considered solved. In the course of experiments, no great success at-

tended the use of more than 25 per cent domestic flake graphite in crucible graphite mixtures. It was, therefore, fitting that the Bureau of Mines should thoroughly investigate not only the use of domestic flake in crucible manufacture, but also the methods of manufacturing crucibles from such graphite.

The future of the graphite industry and the effect of improvements and changes in the brass and steel industries have been well outlined by Ferguson.[a] If new and increasing uses can be developed for graphite, it may be possible through the manufacture of graphite articles of commerce in the various mining districts to establish the domestic mining industry on a firmer basis than is possible with many small operating units that produce only crucible stock. The by-products, No. 2 flake and graphite dust, which are drugs on the present market, would then be of value. The graphite-mining industry made a hearty response to the market for crucible grades created by the request of the War Industries Board, and it would be unfortunate if, with the reestablishment of peace, this industry should revert to its prewar basis.

GRAPHITE INDUSTRY IN ALABAMA.

PLANTS.

There are 39 plants in the three graphite-producing counties of Alabama: Clay, Coosa, and Chilton. About October 1, 1918, four of these plants were in course of erection and fourteen were temporarily closed because of changes in method of treatment or because of plant fires. A little over half of the plants were then operating, and only six were running full time. After the cessation of hostilities production fell rapidly, and in June, 1919, probably not more than half a dozen plants were in operation.

As this industry developed chiefly during 1917 and 1918, operating difficulties still occur.

The plants enumerated have actual operating capacities ranging from 4 to 15 tons an hour, but the rated capacities are about double these figures.

ORE MINED AND METHODS OF MINING.

The graphite occurs in the Talladega schist and is accompanied by quartz, feldspar, mica, other accessory minerals, and alteration products. Only the upper 30 to 60 feet of the graphite schist is mined. This consists of decomposed and weathered schist and is normally rather soft and easily broken, while the unweathered schist or

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[a] Ferguson, H. G., Graphite in 1917: Mineral Resources U. S., 1917, U. S. Geol. Survey, 1918, pp. 97–119.

"blue rock" is comparatively hard and contains considerable pyrite. For this reason, operators are loath to work the "blue rock." Alabama ore contains, as far as is known, no amorphous graphite.

The ore is covered with 1 to 6 feet of overburden. This carries some graphite, but contains so much clay and vegetable matter that it is removed and dumped as waste to obviate milling difficulties caused by the presence of these materials. The stripping is done with plows, scrapers, and wheelbarrows, and does not involve much labor or expense.

All of the companies have open-pit workings. Machine drills are employed at only one mine, where self-rotating hammer drills of the jackhammer type are used to drill holes 15 feet in depth. All other drilling is done by means of a "jumper" drill, holes being drilled 8 to 30 feet deep. All blasting is done with black powder. The deeper holes are chambered with dynamite or blasting gelatin.

The broken ore is loaded into cars by hand, or by steam shovels with a dipper capacity of five-eighths of a cubic yard. With hand loading, end-dump or side-dump cars of 1 to 1¾ ton capacity are employed. With steam shovels, side-dump contractors' cars of 4 to 5 ton capacity are used. About one-fourth of the plants in this district are so situated as to necessitate hoisting ore out of the open pit to the mill. At the plants in which the crude ore enters the mill by gravity, hand tramming is employed, except where the open pit is situated at a considerable elevation above the mill; in such plants, boxed chutes with hand tramming or gravity planes are used. Balanced hoisting is used in only three or four plants. At one plant, a narrow-gage railroad transports ore from the open pit to the mill.

The absence of enough storage room at the head of the mill or at some point between the open cut and the mill is practically general in the district. Only a small proportion of the plants have storage capacity sufficient to permit the accumulation of enough ore to run one shift of 10 hours, the majority of plants having storage capacity to enable milling to continue only for a half shift. Mining is carried on in the open and is dependent, to a certain degree, on the weather. Shut-downs in the mills, owing to operating troubles, are frequent. The result, therefore, of the lack of storage is a loss of operating time, the elimination of which would soon pay for the installation of adequate storage. This point can not be too strongly urged for these small, and, mostly, single-unit plants.

The ore in Alabama averages about 2½ per cent graphitic carbon. From this is obtained generally three products—No. 1 crucible flake, No. 2 flake, and dust. The aim in concentrating is to recover as much as possible of the No. 1 flake, analyzing 85 per cent graphitic carbon and remaining on a No. 8 silk cloth of 86 mesh. No. 2 flake, analyz-

ing 75 to 80 per cent graphitic carbon and of finer size than the No. 1 flake, is a by-product grade for which the market is limited. The dust analyzes 30 per cent or more graphitic carbon and is a greater drug on the market than the No. 2 flake. Hence, the problem of ore dressing is complicated by the necessity of recovering as much as possible of the graphite in the ore in such form as to be marketable as No. 1 flake. The measure of efficiency, therefore, in graphite milling is not the percentage of recovery but the number of pounds of No. 1 flake recovered per ton of ore.

Unfortunately graphite plants do not make a practice of analyzing their mill heads. It is impossible therefore, to establish a relation between the total content of graphite in the ore and the amount recovered as No. 1 flake. Average figures as to the actual production per ton of ore are stated on page 27.

The by-product grades can readily be used in the manufacture of lubricating flake, paint stock, foundry facings, stove polish, etc., but the demands of plants manufacturing these products are not large and the market does not absorb all the No. 2 flake and dust produced.

CRUSHING METHODS.

Many different crushing machines and combinations are used in Alabama in the dressing of graphite ore. The coarse crusher most commonly used is a jaw crusher of the Blake or Dodge type that reduces the ore to about $1\frac{1}{2}$ inch size. In some plants, a toothed impact roll is used for coarse crushing. In plants where dry material is crushed fine, rolls, dry mullers, or Symons disk crushers are used to reduce the material to final treatment size which is about $\frac{1}{16}$ or $\frac{3}{32}$ inch. For fine crushing in plants in which an oil-froth flotation method of concentration is used, coarse crushing is followed by some type of rotating mill, ball mill, pebble mill, or rod mill. These machines are sometimes preceded by a Symons disk crusher.

CONCENTRATION METHODS.

There are four methods of concentrating graphite ores, water "skin-flotation," the pneumatic process, the log-washer process, and oil-froth flotation.

The extent to which these various processes are used in Alabama is as follows, the figures including plants that contemplate putting in those systems under which they have been classified: The skin-flotation washer system is used in twelve and one-half plants. One of the plants consists of two units, one of which is the Minerals Separation oil-froth flotation. The pneumatic system is used in three plants, the log-washer system in two plants, and oil-froth flotation systems in twenty-one and one-half plants.

WATER "SKIN FLOTATION."

In the water "skin flotation" system, a typical flow sheet of which is shown in figure 1, the ore is crushed dry to $\frac{1}{16}$ or $\frac{3}{32}$ inch. It is

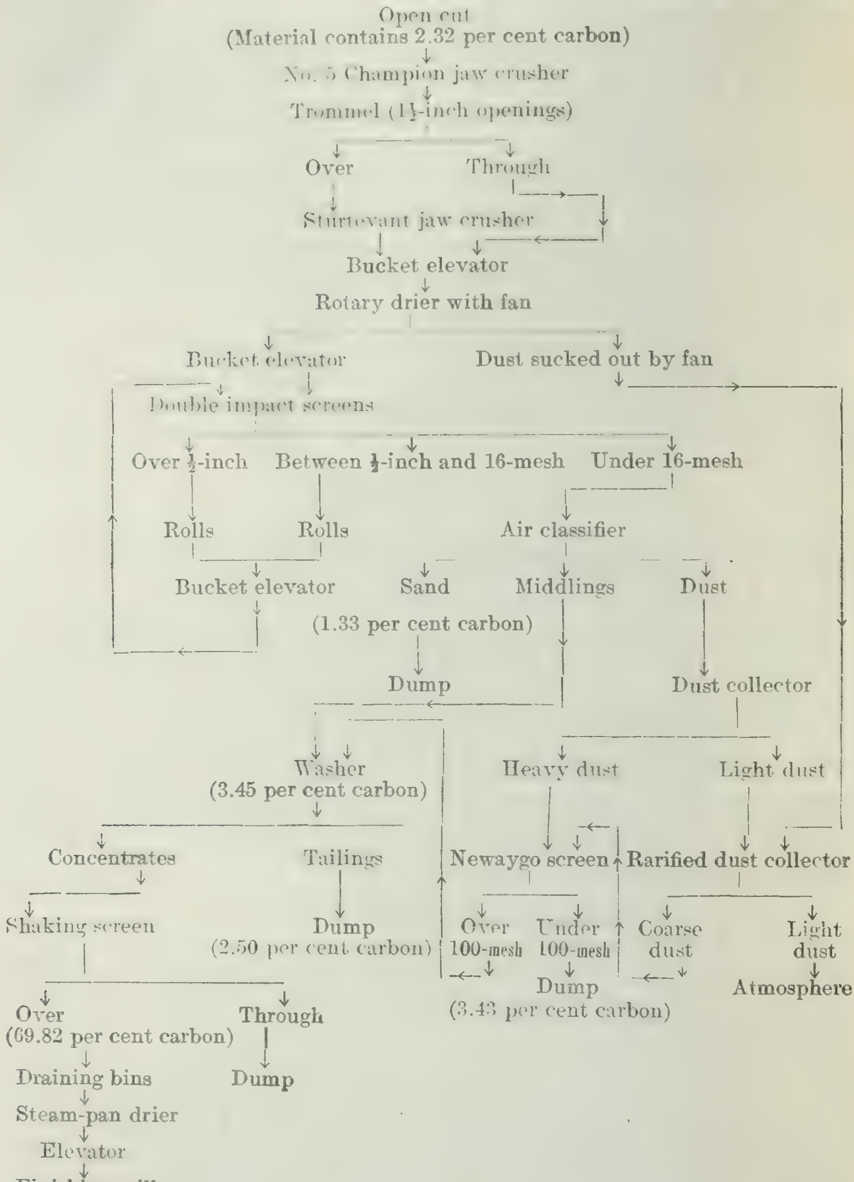

FIGURE 1.—Flow sheet of water "skin flotation" system.

then sent through a rotary drier, screened, and passed over an air separator where it is classified into three products—"scalpings" or

tailings, that go to the waste dump; middlings, that form the "washer" feed; and fines, that are generally treated on a separate group of "washers." The middlings plus the fines, which form 50 to 65 per cent of the crude ore, go to a bin for fine ore, and are fed to "surface-tension" washers where the graphite and some of the mica are floated off, on the surface film of water. The gangue minerals are wet by the water, sink to the hutch of the washer, and are discharged to the tailings launder. The rough graphite concentrate is then dewatered on a trommel or shaking screen, which also removes both the graphite too fine to make No. 1 flake and the fine sand that has been carried over into the concentrates by the film formed by the graphite on the surface of the water. The dewatered concentrate, a dry sample of which analyzes 40 to 60 per cent graphitic carbon, is then dried in a rotary or steam-pan drier and sent to the finishing plant. Average figures for the amount of rough concentrate recovered per ton of ore are not available, but depend of course in large measure upon the grade of ore and of concentrate.

The types of screening and air-classifying apparatus used differ widely, and many of them are unique. Newaygo and "whip-tap" screens are most popular, but other types of impact and shaking screens are used, as well as hexagonal trommels. Most of the air-classifying apparatus is individually designed and erected, and gives results that seem to be efficient. Practically nothing has been done to check up the work of this class of machine; its operation is judged entirely by looking at the products without having even the "scalpings" analyzed. So far as is known, no sufficient quantity of No. 1 flake would be recovered to justify additional treatment, with a view to obtaining more No. 1 flake from these "scalpings."

The result of sampling crude ore, or crude concentrates, and "scalpings" is shown in the table following. All samples were analyzed at the Pittsburgh experiment station of the Bureau of Mines.

Results of operation of air classifier.

[Figures represent percentages of graphitic carbon.]

Heading.	Graphitic carbon, per cent.	Graphitic carbon in "scalpings" or tailings, per cent.
Operating on crude ore:		
First sample	3.45	1.35
Second sample	2.55	1.01
Operating on crude concentrates:		
First sample	62.80	4.68
Second sample	48.80	1.83

There are three types of "skin flotation" washers sufficiently distinct in principle to permit individual description. Figures 2, 3, and 4 illustrate the underlying principles in design. The dimensions given are estimates and not actual measurements.

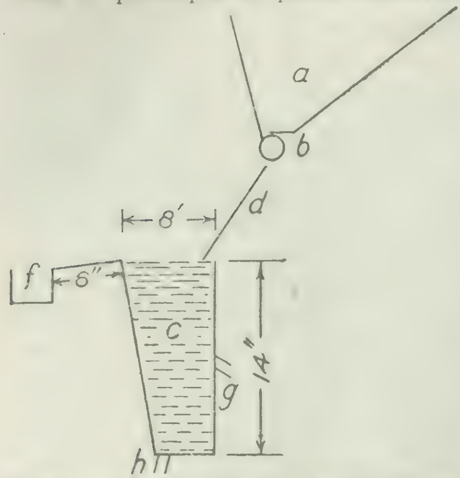

FIGURE 2.—Section through original Munro washer (dimensions indicated are approximate). *a*, Stock bin; *b*, feed cylinder (2-inch revolving shaft); *c*, lake 8 inches wide; *d*, baffle inclined 50° to 65° from horizontal, 10 inches long; *f*, collecting launder, into which graphite is carried over an apron from *c*; *g*, water inlet; *h*, hutch spigot.

MUNRO WASHER.

The Munro washer is a rectangular washer in which the feed drops from the feed cylinder to an inclined flat baffle and thence to the water film that carries the graphite to the collecting launder and permits the gangue to sink to the hutch. These washers are built in units about 4 feet long, placed end to end and back to back.

COLMER WASHER.

The Colmer washer is a circular washer with a conical feed baffle. The ore falls from the baffle on a revolving circular disk with ribs cast on it. The tangential motion thus imparted to the ore is intended to assist in carrying off the graphite by reducing the tendency of the falling particles to rupture the surface film of water. Some operators claim that better results are obtained with this washer by keeping the disk stationary, relying on the disk solely to change the direction of travel of the particles of ore. These operators claim that the tangential motion given by the revolving disk produced a lower-grade concentrate than was obtained by keep-

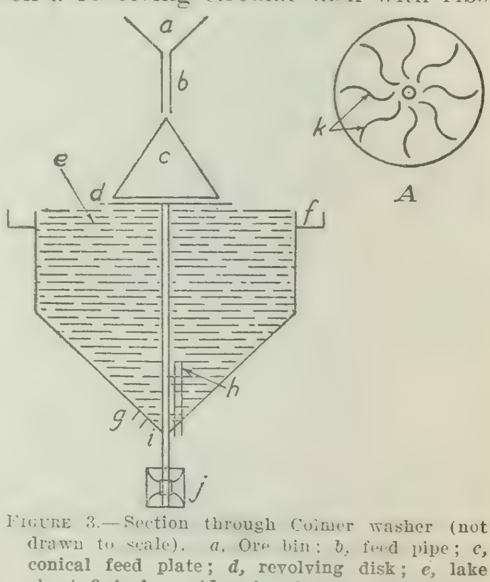

FIGURE 3.—Section through Colmer washer (not drawn to scale). *a*, Ore bin; *b*, feed pipe; *c*, conical feed plate; *d*, revolving disk; *e*, lake about 9 inches wide; *f*, collecting launder; *g*, hutch spigot; *h*, water inlet; *i*, shaft; *j*, pulley for rotating disk; *k*, ribs. A, detail of disk *d* (about 18 inches in diameter).

ing the disk stationary. As shown in figure 3, the washer is about 42 inches in diameter.

NEW MUNRO WASHER.

The new Munro washer (fig. 4), which also is circular in shape, is 6 to 12 inches larger in diameter than the Colmer, most of this increase in size being taken up by increasing the width of the "lake." In the Munro washer, the treatment water is forced through an annular opening, formed by placing a cast-iron cover over a bell-mouth casting which is attached to the feed-water pipe. This washer does away with the mechanical motion of the Colmer type. It is difficult, however, to keep an unobstructed flow of water through the annular opening.

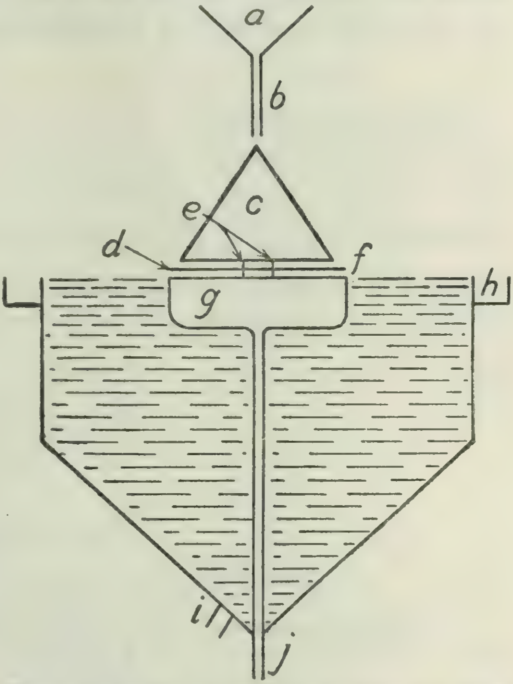

FIGURE 4.—Section of new Munro washer (not drawn to scale; lake about 12 inches wide). *a*, Stock bin; *b*, feed pipe; *c*, conical feed plate; *d*, cover plate; *e*, set screws for regulating width of opening; *f*, annular opening; *g*, bell mouth; *h*, collecting launder; *i*, hutch spigot; *j*, feed-water pipe.

PNEUMATIC PROCESS.

In the treatment of graphite ores by the pneumatic process, pneumatic apparatus is used throughout. A typical flow sheet for this system is not given, because the system has been successful in only one mill. The details of the operation of this plant can not be made public.

LOG-WASHER PROCESS.

A typical flow sheet of the log-washer process is shown in figure 5. In this method of treatment the crude ore is crushed and screened without drying. It is then treated in log washers, kerosene oil being added to the water in the apparatus. The concentrates are screened, washed on a cement floor, drained, and dried. This system eliminates the expensive preliminary drying of the crude ore, necessary with the water "skin flotation."

The simplicity and effectiveness of this plant are clear to the observer. The grade of rough concentrate made is excellent and the only important loss occurs in the coarse-sand tailings, which with

regrinding might be made to yield more flake graphite. A test of the coarse-sand tailings, in the same way that has been suggested under the discussion with reference to the "scalpings" of air classifiers, would determine the desirability of regrinding. The fine-sand tailings are remarkably free from detached flakes of graphite.

OIL-FROTH FLOTATION SYSTEMS.

The majority of the graphite plants in Alabama have installed some type of oil-froth flotation cell. Eight plants have installed the Callow pneumatic cells, four have installed the Simplex type of cell, three and one-half (a two-unit plant has one skin-flotation

Ore bin
↓
No. 5 Champion jaw crusher
↓
Dry muller ($\frac{3}{32}$-inch screen)
↓
Bucket elevator ← Add water
↓
Distributing screw conveyor ← Add kerosene oil
↓
Four sets of log washers of two each (3.31 per cent carbon)

- Tailings (1.09 per cent carbon) → Dump
- Concentrates → Three hexagonal trommels
  - Through → Dump
  - Over (31.73 per cent carbon) → Concrete washing floor
    - Heavy material (sandy) → Dump
    - Light material (concentrates: 58.8 per cent carbon) → Draining bin → Steam-pan drier

FIGURE 5.—Flow sheet of log-washer process.

washer system) have installed the Minerals Separation cells, and six plants have installed, or intended to install, homemade cells or washers combining some of the principles of these processes.

The chief difficulty with oil-froth flotation systems is in the loss of the large flake. In recovering the large flake, so low grade a concentrate is obtained that an added burden is placed on the finishing plant. There is no difficulty in obtaining a high recovery of the high-grade dust, but there is at present no ready market for this character of material. The best results obtained in the district are from the homemade cells or washers mentioned.

In both the Minerals Separation and the Callow systems, fine crushing is accomplished by Marcy, Lehigh, Marathon, or Hardinge ball or pebble mills. In practically all of the plants in which these two systems have been installed, water classifiers of the Deister or Gemmell type are used. Two plants have Dorr classifiers. For dewatering the rough concentrate, vacuum filters of the Portland or Oliver type are generally used rather than shaking screens.

MINERALS SEPARATION SYSTEM.

A typical flow sheet of the Minerals Separation system is shown in figure 6. Assay results for this system are not available.

CALLOW PNEUMATIC SYSTEM.

A typical flow sheet of the Callow pneumatic system is shown in figure 7. This system has received greater development in the Alabama district than any other oil-froth flotation system. At one plant the tailings from the Callow cells are treated in an improvised washer with satisfactory recovery.

Samples obtained in Callow plants did not make a good showing, chiefly because of the physical difficulties in taking them. The results of sampling follow:

Results of operation of Callow pneumatic cells.

[Figures represent percentages of graphitic carbon.]

Test.	Crude ore.	Crude concentrates.	Tailings.
No. 1	2.24	43.66	Sample "salted."
No. 2	Sample lost	52.59	No samples possible.
No. 3	No sample taken [a]	57 to 80	0.5 to 0.9.

[a] At this plant the flake seemingly is being subjected to excessive grinding, with the production of a large quantity of graphite, which will not produce No. 1 flake.

SIMPLEX SYSTEM.

The Simplex system includes crushing as well as concentration. The crushing apparatus has no particular advantage over other crushing machinery but the concentration method appears to be efficient.

In this system, the pulp comes unclassified to the center of an elliptical washer on the surface of which jets of water, with flotation oils, are forced at a pressure as high as 40 pounds per square inch. The jet of water entrains air, and a froth is formed which floats the graphite over the side of the washer, while the gangue drops to the hutch.

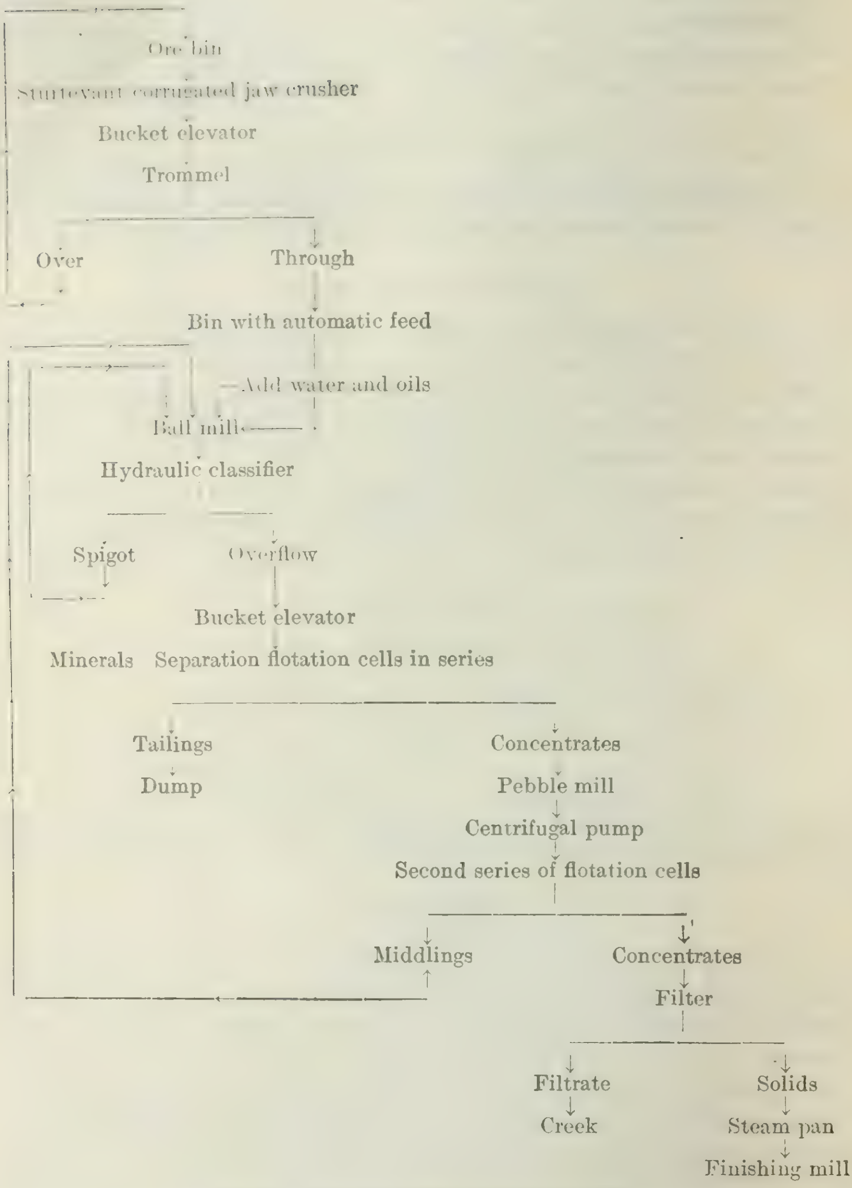

FIGURE 6.—Flow sheet of Minerals Separation system.

Ore bin
↓
Toothed-roll crusher
↓
Shaking screen (3/8 inch opening)

Over | Through
↓
Bucket elevator
↓
Symons disk crusher
↓
Bucket elevator
↓
Fine-ore bin with automatic feed regulator
↓
Belt conveyor ——— Add water and oil
↓
Shaking screen (12-mesh)

Over | Through
↓ | ↓
Fine rolls | Deister classifier
↓
Bucket elevator (returns to shaking screen)

Spigot | Overflow
↓ | ↓
Dump | Callow "rougher" cells

Concentrates | Tailings
↓ | ↓
Callow "cleaner" cells | Dump

Tailings | Concentrates
↓
Shaking screen

Over (52.59 per cent carbon) | Through
↓ | ↓
Steam pan drier | Dump
↓
Refining plant

FIGURE 7.—Flow sheet of Callow pneumatic system.

A typical flow sheet for a plant of this type is shown in figure 8, and a sketch of a section through the washer is shown in figure 9.

In various mills in Alabama, Wilfley and Deister tables are being installed to raise the grade of the rough concentrate and to remove gangue from the concentrate fed to the finishing plant. The gangue wears the surfaces of the buhr stones, thus necessitating their more frequent dressing, an operation that is both tedious and expensive.

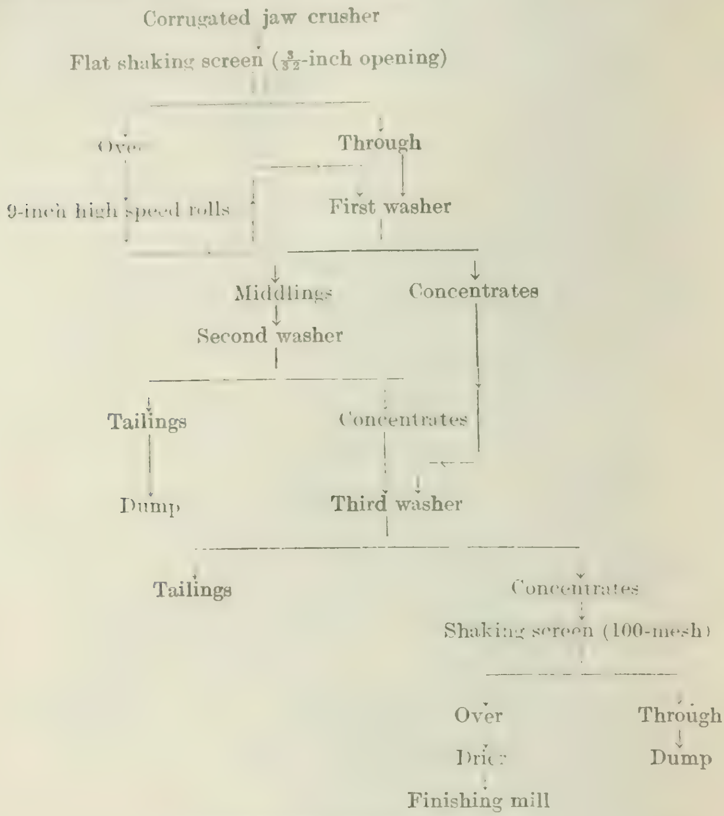

FIGURE 8. Flow sheet of Simplex system.

At one of the plants, concentrates from a table are treated in an improvised pebble mill. The dust is then removed, and the material is passed through a series of screens, producing No. 1 flake ready for market.

The most important consideration as regards the use of concentrating tables is their capacity. As the use of tables in this district is still in the experimental stage, reliable data as to the success of this new practice is not available.

REFINING METHODS.

The rough concentrate as it comes from the concentrating plant contains a minimum of 40 per cent graphitic carbon. In some plants, this crude concentrate analyzes as high as 75 per cent graphitic carbon, although the usual proportion is 40 to 60 per cent. The impurities are quartz, mica, wood fiber, etc. Material of this character is not readily marketable except at prices far below those obtained for No. 1 flake; moreover, the market for crude concentrate is limited. These conditions, therefore, necessitate the refining of crude concentrate to No. 1 flake, containing 85 per cent graphitic carbon, and remaining on a No. 8 silk cloth of about 86 mesh.

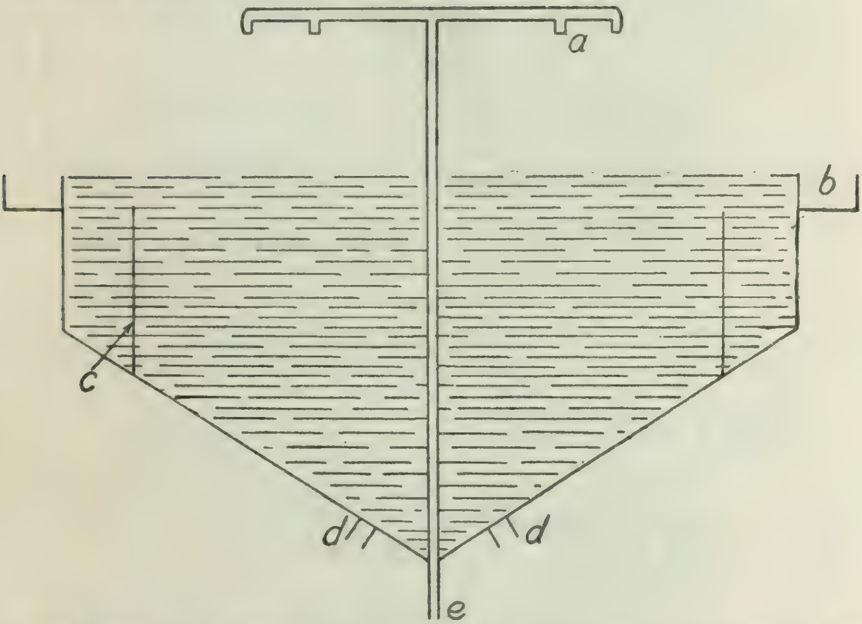

FIGURE 9.—Section of Simplex washer (not drawn to scale; major axis is about 6 feet and minor axis about $3\frac{1}{2}$ feet). *a*, Spigots, $\frac{1}{4}$ to $\frac{3}{8}$ inch orifice, water and oil under pressure; *b*, collecting launder; *c*, screen baffle; *d*, hutch spigots; *e*, feed water.

Microscopic examination shows that the graphite flakes consist of thin laminae of graphite and that present between the laminae are associated minerals, chiefly quartz and mica, which must be eliminated. There are two general methods of refining:

(1) If the crude concentrate comes to the finishing plant with most of the impurities still interlaminated with or attached to the graphite, grinding in a pebble or buhr mill is necessary in order to loosen the graphite from the impurities.

(2) On the other hand, if the crude concentrate comes to the finishing plant with the larger part of impurities as detached par-

ticles, refining can be done pneumatically or with electrostatic machines. Where this method is employed, crushing in the concentrating plant has been of such a character as to accomplish what is usually done in the finishing plant with the buhr or pebble mill.

The first method is the one most generally used; the second method is used in only two plants, and its application is not broad enough to warrant more detailed description at this time.

A flow sheet of a refining plant using the first method is shown in figure 10. This flow sheet is not typical because most plants are

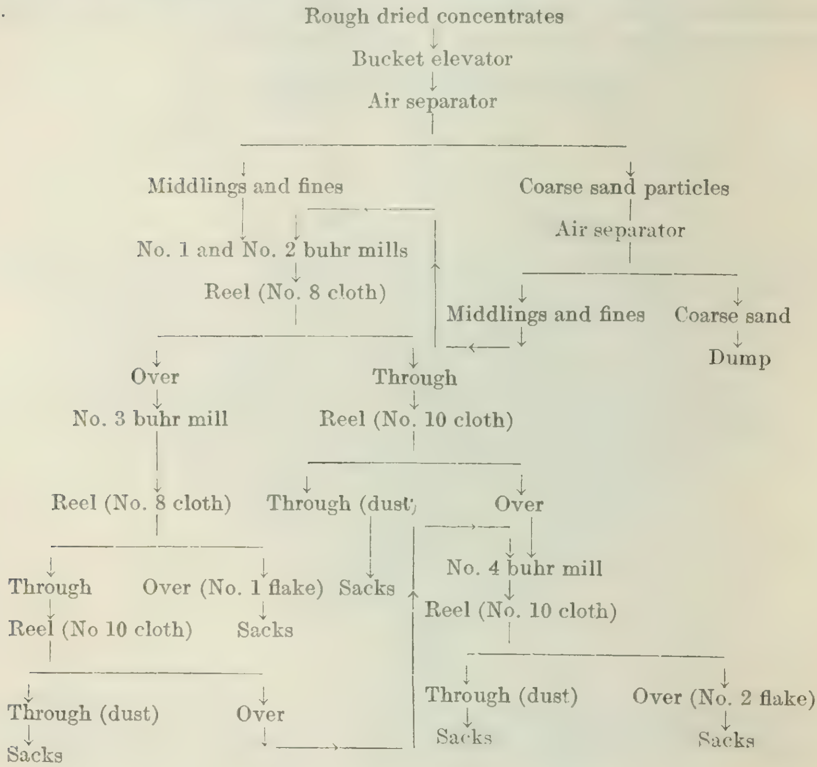

FIGURE 10.—Flow sheet of graphite refining plant.

quite dissimilar, but it represents in a general way the underlying principles governing refining.

AIR CLASSIFICATION.

Air classification is used in most of the refining plants and forms an important step in the refining process. Figure 11 shows sketches of air classifiers. Such apparatus is usually improvised. So far as is known no experimental work has been done by the large majority of operators with a view to determining the most efficient proportions and shape for this device.

In principle air suction created by a fan at the back end of each chamber is applied to a falling stream of graphite ore or concentrate. The heavier material, quartz grains with attached graphite, is practically unaffected by the air suction, and falls into the hopper farthest from the fan. The lighter particles are drawn nearest the fan, which sucks the finest dust into the atmosphere. When this

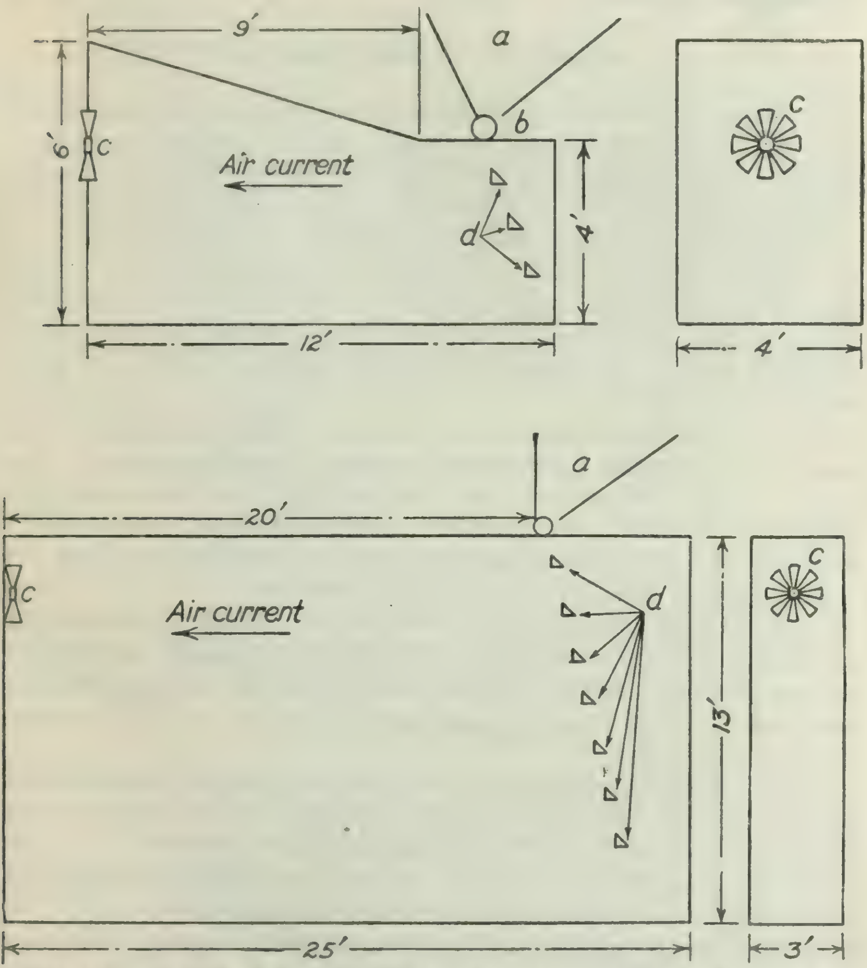

FIGURE 11. Two types of air classifiers (not drawn to scale). *a*, Stock bin; *b*, feed shaft; *c*, fan; *d*, baffles. Three sets of hoppers are provided for the three products usually made.

apparatus is used on crude concentrates, the middlings—which include the heaviest flake—form No. 1 flake stock, going to the No. 1 buhr mills: the finer material nearest to the fan is screened to remove as much finished No. 1 flake as possible.

In all refining methods the following steps are taken to facilitate the highest possible recovery of No. 1 flake:

1. All dust is eliminated from the circuit as soon as practicable.

2. No. 1 flake is removed from the system as soon as it has been made.

3. As little concentrate as possible is sent to the grinding apparatus.

For analyses of products see discussion of air classification under "Concentration Methods," page 15.

RECOVERY OF FINISHED PRODUCTS.

In discussing recovery in graphite plants it should be pointed out that the individual plants are small mills in which systematic sampling and weighing of products are not regularly employed. No data, therefore, are available as to the extraction obtained at various stages of concentrating and refining. It is impossible, also, to state where the greatest losses occur in these operations except from observation and rough sampling in the field. The results of this sampling are indicated wherever practicable on the flow sheets presented herein; the points at which the greatest losses occur have also been indicated.

The only products actually weighed and sampled are the finished products. It would be entirely feasible to sample regularly the crude ore, the tailings from the roughing mill, and the crude concentrates. Various other products could be sampled from time to time, and this work would amply repay the trouble of sampling.

Recoveries of finished materials are as follows:

1. No. 1 flake analyzing 85 to 90 per cent graphitic carbon and remaining on a No. 8 (86-mesh) or a No. 12 (125-mesh) silk cloth—the smaller flake being obtained when a 90 per cent graphite is produced: 10 to 30 pounds is obtained per ton of ore. The average is about 19 pounds.

2. No. 2 flake, analyzing 75 to 80 per cent graphitic carbon and passing through the meshes above mentioned: 2 to 15 pounds is obtained per ton of ore. The average is about 5 pounds.

3. Dust, analyzing 30 per cent or more in graphitic carbon: An average of about 3 pounds per ton of ore is obtained.

If there were a market for No. 2 flake and dust, a greater recovery of these materials per ton of ore could be made.

The recovery percentages for all products are shown in the table following, which represents average milling practice in the district:

Data showing average recoveries of graphite products in Alabama mills.

Material.	Carbon.	Weight.	Weight multiplied by per cent.	Recovery.
	Per cent.	*Pounds.*		*Per cent.*
No. 1 flake	87	19	1,653	33.06
No. 2 flake	77	5	385	7.70
Dust	30	3	90	1.80
Total finished products	....	27	2,128	42.56
Tails (by difference)	1.46	1,973	2,872	57.44
Crude ore	2.5	2,000	5,000	100.00

The amount of No. 1 flake produced per man per hour ranges from 4½ to 8 pounds, and averages about 6 pounds, figured on the total number of men and officials employed. Proportionate quantities of No. 2 flake and dust are obtained. There is no definite advantage apparent from figures obtained from the different classes of plants. The outputs, of course, depend to a large extent on the amount of graphite originally present in the ore, so that although these figures are an index of costs, they can not be used as a measure of comparative efficiency.

COMPARATIVE COST OF ERECTING PLANTS.

The cheapest type of plant to install is that using a log washer, and this is probably the cheapest to run. No figures are available as to either initial or operating costs for a plant of this kind.

For a plant capable of treating 10 tons per hour, the initial cost ranges from $35,000 to $60,000 erected, not including that of a finishing plant, which would add $5,000 to $10,000 more to the cost.

FIELD SAMPLING.

In the flow sheets accompanying this report, available assays of samples taken in the field have been inserted, and these give some idea as to the relative efficiency of different methods of milling and of steps in the various processes. In many instances where assays have been omitted, no samples were taken; and sometimes the samples taken were lost either in transit or in drying in the field, or the assay results were inconclusive because of the difficulty of getting a representative sample in the plant.

GRAPHITE INDUSTRY IN NEW YORK.

PLANTS.

The mining of graphite in New York was started about 60 years ago. This State for many years produced more graphite than any

other, but since 1915 has yielded first place to Alabama, where the growth of the industry has been remarkably rapid.

In 1918 there were three mines in operation, namely, the mine of the Graphite Products Corporation, 8 miles north of Saratoga Springs; Hooper Bros. mine, 4 miles west of Whitehall; and the mine of the American Graphite Co., worked by the Joseph Dixon Crucible Co., 4 miles west of Hague.

OCCURRENCE OF ORE.

In New York the ore averages 4 to 6 per cent graphitic carbon as compared with 2½ per cent in Alabama. There are two important types of graphite deposits in the Adirondack foothills of New York—those on the contact of a limestone and pegmatite, and those on the contact of the Hague garnet-sillimanite gneiss with the Faxon limestone or Swede Pond gneiss. In deposits of the former type, large flakes of graphite are obtained, but such deposits are extremely pockety, and their development has not been profitable. Present production is from the second type of deposit, in which the ore is quite regular and is of a more uniform grade.

The ore on the whole is much harder and more siliceous than the Alabama ore and resembles in many respects the unweathered "blue rock" of Alabama, the original bluish Talladega schist.

MINING METHODS.

Graphite ore is mined in New York both in open pits and underground. The larger part of the ore comes from underground mining. All drilling is done with power drills. On account of the siliceous character of the ore, a greater quantity of fines is produced during crushing than is obtained in Alabama. The ore-bearing zone is usually not over 25 feet thick and averages about 15 feet.

OPEN-PIT MINING.

Wherever possible, the open-pit system of mining is used because of the ease and cheapness of preparing an open pit, where there is no excessive amount of overburden that requires stripping.

UNDERGROUND MINING.

The deposits dip 25° to 35° so that it soon becomes necessary to go under cover for the ore in the course of the development of any mine. This has its advantage in the North because of the long and severe winters which hamper open-cut work.

The system of underground mining at the largest mine in the district is the room-and-pillar method with underhand stoping. In new developments in the district, a definite system of mining has not as yet been determined.

CONCENTRATING METHODS.

A flow sheet of one of the plants in New York is given in figure 12. In this plant gravity stamps are used as fine crushers and "buddles" as concentrating machines. Concentrating tables are used for treating "buddle" middlings, whereas, in Alabama they are to be

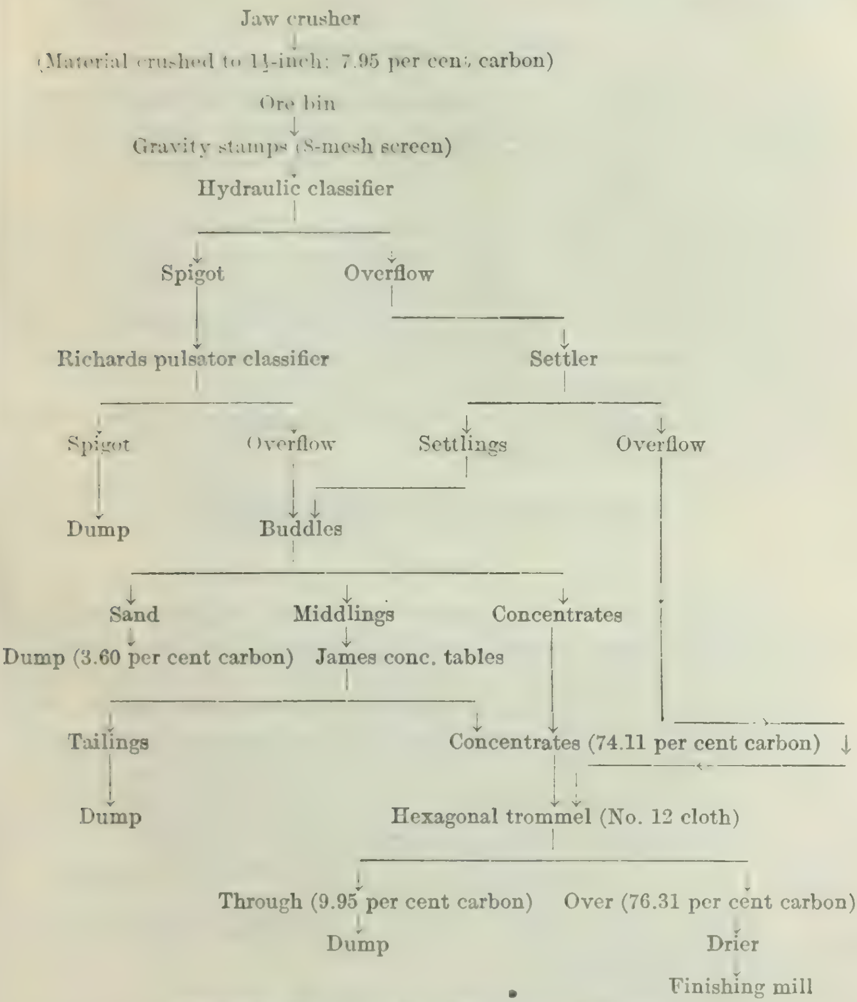

FIGURE 12.—Flow sheet of New York concentrating plant.

employed in raising the grade of rough concentrates preparatory to treatment in the finishing mills.

With crude ore that analyzed 7.95 per cent graphitic carbon, "buddle" tailings analyzed 3.60 per cent C. Tailings from various other machines analyzed 3.96 to 9.95 per cent C., the latter product

consisting of the fines through the rough concentrate trommel. The crude concentrate analyzed 76.31 per cent graphitic carbon.

It should here be noted that the "buddle" is being replaced at the Jos. Dixon Crucible Co. plant by a system of oil flotation. As the product from this mine goes into the manufacture of materials other than crucibles, it is desirable to get as high a recovery of graphite of various grades as possible per ton of ore.

REFINING METHODS.

Refining methods do not differ greatly from methods used in Alabama plants. One variation is the use of the Hooper air jig instead of air classification. The chief disadvantage ascribed to this jig is its small capacity.

The American Graphite Co. refinery at Ticonderoga prepares foreign and domestic graphite, both amorphous and crystalline, for use in the manufacture of commercial articles, such as paints, foundry facings, pencils, lubricating flake, and boiler compounds. The by-products obtained from this mill are all utilized in the manufacture of the commodities mentioned.

Data as to recoveries of No. 1 flake, No. 2 flake, and dust in New York practice are not available.

THE INDUSTRY IN PENNSYLVANIA.

PLANTS.

The graphite industry in Pennsylvania has in the past suffered from "wild-cat" and "get-rich-quick" schemes that for a time seriously interfered with the production in the Pickering Valley, Chester county. During the war, this district, in common with other producing districts, responded to the increased demand for graphite. In 1918, five plants were in operation, two close to Byers and three near Chester Springs. The most recent installation is the Huff electrostatic system at the plant of the Rock Crucible Graphite Co., the contemplated flow sheet of which is given in figure 13.

At present (August, 1919) graphite production in Pennsylvania is practically at a standstill.

OCCURRENCE OF ORE.

The ore, with one exception, is a soft disintegrated schist dipping from 35° to 50° and analyzing $3\frac{1}{2}$ to $4\frac{1}{2}$ per cent graphitic carbon. In some places most of the gangue has been removed by weathering, the graphite remaining behind as large coarse flakes.

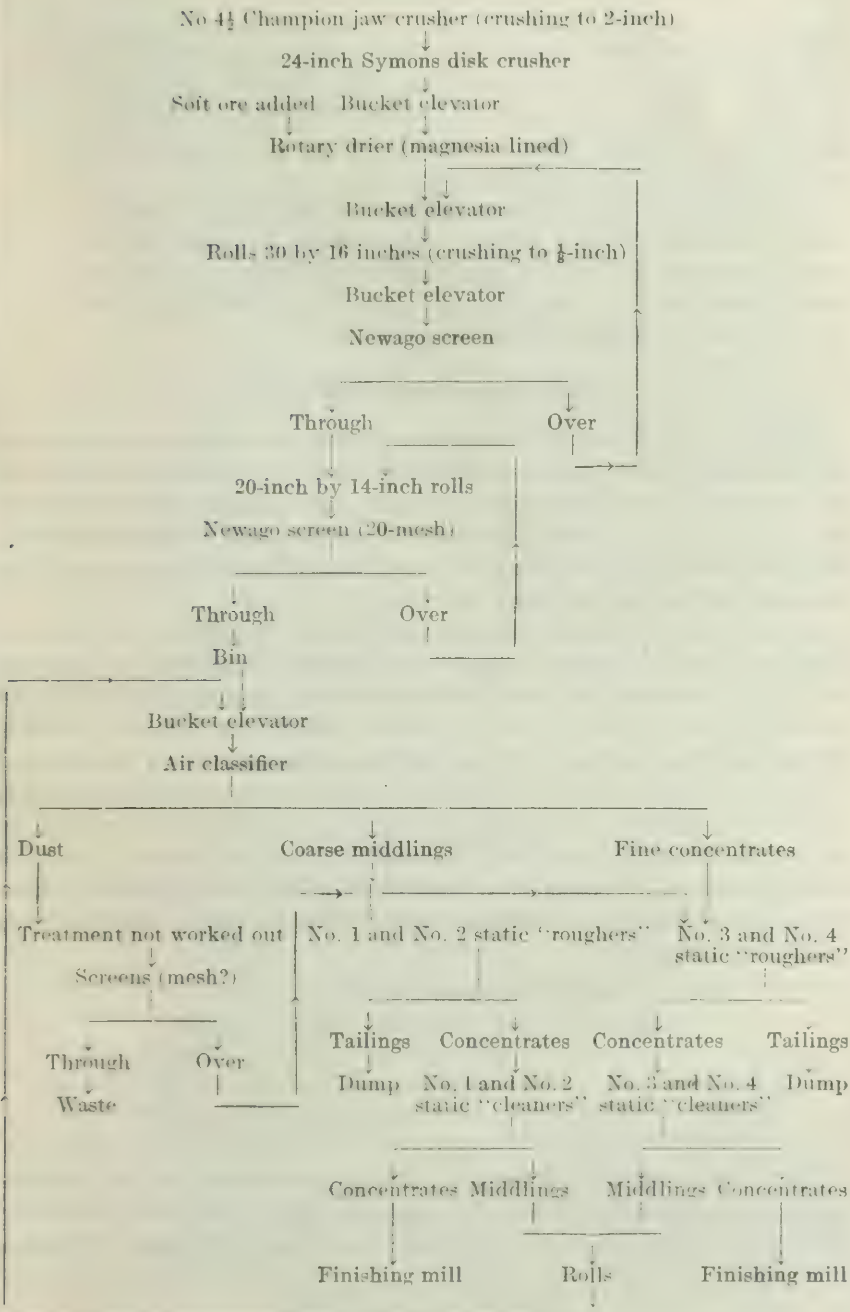

FIGURE 13.—Contemplated flow sheet for Huff electrostatic system.

MINING METHODS.

With the exception of the operations of the Standard Carbon Co. and part of the operations of the Rock Crucible Graphite Co. all mining is done in open pits. Loading of broken ore is done either by hand or by steam shovel, and little or no drilling is necessary because of the character of the ore.

At the mine of the Standard Carbon Co. the ore is mined in narrow rooms with alternating pillars. The deposit averages 25 feet thick and dips about 40°. The mine is developed by a 120-foot vertical shaft, with levels at 80 feet and 120 feet below the collar. The ore is readily drilled with an auger drill.

CONCENTRATING METHODS.

In this district three of the concentrating systems described under the outline of the Alabama industry are used. Three plants use the log washer or a similar device; one plant is using the dry process in the form of the Huff electrostatic system, and a fifth plant was using the oil-froth flotation system with the K and K type of cell, but has now discarded this type of cell.

At two of the plants a machine called the "oscillator" is used in place of the log washer. The "oscillator" comprises an inclined table with a series of rakes suspended over it and attached to an eccentric. In addition to the horizontal motion each row of teeth is given a circular motion in a vertical plane, thus stirring the pulp and releasing the graphite, which floats to the lower end of the table while the tailings are worked to the upper end by the rakes. In principle, therefore, this device resembles the log washer. As with the log washer, kerosene oil is used to help float the graphite. Samples taken in plants in which machines of this kind are used gave the following results:

Results of operation of oscillators.

[Figures represent percentages of graphitic carbon.]

	Test 1.	Test 2.
Crude ore	4.52	3.56
Washer tailings	2.19	1.79
Rough concentrates	62.00	72.07

The contemplated flow sheet of the plant in which the Huff electrostatic system has been installed is shown in figure 13. The other four plants have flow sheets similar to those described under Alabama practice.

REFINING METHODS.

Methods of refining used in the Pennsylvania field are similar to those in use in other districts. Two refining plants are being oper-

ated. In the new Huff electrostatic plant it is expected that no additional refining apparatus except dusting and bolting screens will be required. The plants not operating refineries sell their rough concentrate, representing material coarser than 100 mesh and analyzing 60 to 70 per cent graphitic carbon, to an Eastern refiner.

In this district data as to the recovery of finished products are not available. From 50 to 60 pounds of rough concentrate, analyzing as shown in preceding table, are recovered per ton of ore.

THE INDUSTRY IN TEXAS.

OCCURRENCE OF ORE.

In Texas graphite is found in the siliceous pre-Cambrian Packsaddle schist in the central region of the State between Llano and Burnet. In many places, the graphitic schist has been disrupted by granite and pegmatite intrusions. This schist, according to Paige,[a] is a metamorphosed sedimentary rock in which the carbonaceous materials have been recrystallized to form graphite.

The ore analyzes 6 to 10 per cent graphitic carbon, but the recovery of No. 1 flake is comparatively low because of the presence of amorphous graphite and very small flake in the ore; moreover, the gangue is hard, so that in crushing, a large amount of the flake is destroyed by being ground to finer sizes than are used as No. 1 flake.

In 1918, three properties were in operation in the State, but in June, 1919, only one was active.

MINING METHODS.

At the properties of the Southwestern Graphite Co., 9 miles west of Burnet and the Burnet-Texas Graphite Co., 7 miles west of Burnet, open pits have been developed; but at that of the Dixie Graphite Co., 4 miles northeast of Llano, mining is by underground methods.

At the property of the Southwestern Graphite Co., the overburden, which varies in thickness from 2 to 8 feet, is a hardbaked detritus containing large boulders of ore. It contains much clay and vegetable fiber and is said to average 3 or 4 per cent graphitic carbon, only a small part of which can be recovered as No. 1 flake stock. Accordingly, this material is loosened by means of a plow aided by a snatch block and is dumped as waste. In loosening overburden of this character, blasting powder might advantageously be used.

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[a] Paige, Sidney, U. S. Geol. Survey, Geol. Atlas, Llano-Burnet folio (No. 183), 1912, p. 4.

When the mine was visited the ore body was being crosscut, preliminary to the development of two working faces along the strike of the ore, which is 80 to 100 feet thick and dips about 65° southeast. The outcrop can be followed on this company's property with a slight interruption for almost three-fourths of a mile along the strike.

In mining, a Keystone wood-fired churn drill is used for drilling holes 5½ inches in diameter. These are placed at the corners of 12-foot squares the first row of which is 12 feet from the last crack of the previous blast. All holes are drilled to a depth of 25 or 30 feet, which is 5 feet below the elevation of the bottom of the open pit. Blasting gelatin of 60 per cent strength is used for breaking. An Erie wood-fired steam shovel with a dipper capacity of five-eighths of a cubic yard loads the broken ore into 5-ton side-dump contractors' cars which are hauled to the mill by a Whitcomb gasoline locomotive. Large boulders are placed to one side by the shovel and are block-holed with self-rotating hammer drills of the jackhammer type.

The Burnet-Texas Co.'s property is 2 miles northeast of that of the Southwestern Graphite Co. on the same schist series and at the time of visit was being worked in a small way. Here the ore is more frequently cut by granite and pegmatite intrusions, and as developed appeared to contain less graphite that would make No. 1 flake stock. Besides an open pit, this property was being prospected by a vertical shaft.

At the Dixie Graphite Co. property, 4 miles northeast of Llano, the ore is won by underground mining. The deposit dips to the southeast at a flatter angle than at the Southwestern Graphite Co.'s property. Near the surface the ore contains considerable quantities of amorphous graphite and fine flake. That mined underground is of better grade as regards the size of flake, and contains considerably more pyrite and mica than were obtained in the surface ore. In the past, much development work was done on this property with a view to mining gold and molybdenum ores, but these efforts were not successful.

CONCENTRATING METHODS.

Two plants use the oil-froth flotation method; at a third plant Huff electrostatic machines have been installed. These plants do not differ materially from mills previously described, except that in each a rough concentrate analyzing 70 to 80 per cent graphitic carbon and remaining on an 80 or a 90 mesh screen is recovered.

No refining plants are as yet in operation, although one company is reported to have completed alterations in its mill that will permit production of refined graphite.

In this district the same lack of storage facilities for crude ore was noted as in the Alabama field. The general remarks as to storage also apply to the New York and Pennsylvania districts.

RECOVERY OF GRAPHITE FROM "KISH."

The product known as "kish" is a mixture of graphite, slag, iron oxide, fragments of iron, and other materials that accumulate about a pig-iron casting ladle, or a Bessemer or open-hearth furnace. The graphite is formed during cooling by the crystallization of excess carbon from molten pig iron. The kish obtained for refining has contained 8 to 10 per cent of graphitic carbon in the form of a light, thin, and fluffy flake.

The method of concentrating and refining kish is similar in many ways to the methods employed with natural graphite. The chief points of difference are that kish is not subjected to crushing, but is carefully screened and is treated with magnetic separators. A patent (No. 1239992) on the recovery of graphite from kish has been issued to F. W. Weisman, and another patent (No. 1271146), to E. C. Ewen.

Kish as a possible source of graphite should not be overlooked. It is questionable, however, whether enough of this material can be obtained to interfere or compete seriously with the mining of natural flake graphite.

EXPERIMENTAL WORK ON CONCENTRATION AND REFINING.

As is evident from the foregoing discussion, much work had been done on various methods of concentrating graphite ore and refining graphite, but no large amount of effort had been expended on making comparative tests of the processes. It is evident, too, that the possibilities of recovering larger quantities of No. 1 flake have not been exhausted. For these reasons the Bureau of Mines has felt justified in conducting experiments at its Salt Lake City experiment station on the concentration of graphite ore and the refining of graphite. This work is described in the report of F. G. Moses, "Refining Alabama flake graphite for crucible use" (pp. 46 to 74).

EXPERIMENTAL WORK ON CRUCIBLE MANUFACTURE.

The Bureau of Standards at various times has done some work on crucible manufacture with a view to the use of materials other than the imported clays and graphites. This work has never been carried to a conclusion. It was felt therefore that the Bureau of Mines at its Columbus experiment station should make an investigation that would decide to what extent flake graphite might be used in crucible

manufacture, and would establish physical and chemical specifications for domestic flake graphite.

PHOTOMICROGRAPHY.

As microscopic examination of crucibles made by various manufacturers, in conjunction with a knowledge of the crucible ingredients, may be of assistance in the solution of the mechanical difficulties attending the use of more than 25 per cent domestic flake graphite in crucible graphite mixtures, this work has been assigned to the Pittsburgh experiment station of the Bureau of Mines.

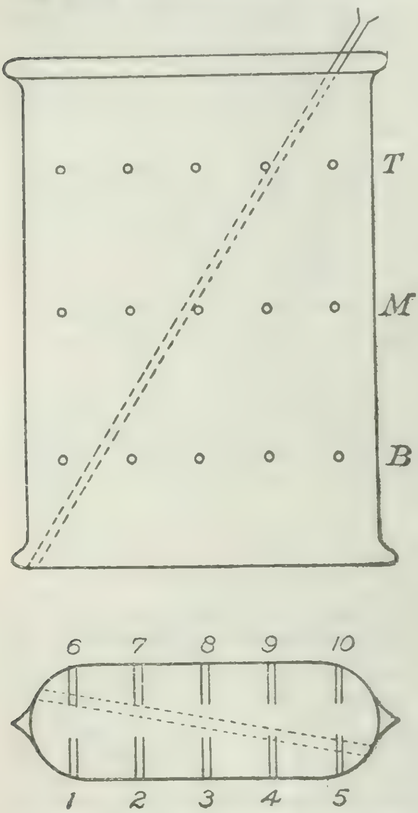


FIGURE 14.—Method recommended for sampling finished sacked product. Correct position of sampling device indicated by dotted lines in two views. The method now generally employed gives samples corresponding to 1 (T, M, B), 3 (T, M, B), or 5 (T, M, B).

METHODS OF SAMPLING.

Generally speaking, the only samples taken in graphite plants are of the finished products. In the past these have not been sampled in the same way by producer and by consumer, so that serious disputes over shipments have arisen, with the result that consumers have often refused to accept certain shipments.

By daily sampling crude ore and crude concentrates, as well as finished products, a much better check on mill work can be obtained than is possible by sampling only the finished grades.

Domestic graphite is usually marketed in sacks holding 150 pounds. The sacks are filled from a spout at the bottom of a small bin to which the products come from the final sizing screen, which follows the buhr mill. In the process of refining, a product absolutely homogeneous in carbon content is not being made at all times of the day. The material that goes to the finished-product loading bin often varies 5 per cent from the standard determined by the mill operator. The causes of these variations are many. Thus, products obtained just after the buhrstones have been freshly dressed are different from those obtained before the stones have been dressed;

and the feed to the buhrstones varies in character from time to time because of frequent concentrator shutdowns. The stones are adjusted from time to time during the day, but sometimes the character of the No. 1 flake obtained is visibly below the predetermined plant standard. As the sack is filled from a vertical spout, any fluctations in the grade of graphite in the bin appear as strata in the bag. Moreover the larger flakes naturally fall to the edges of the pile under the spout. The miller usually samples a bag of graphite by taking a spoonful of graphite from the top of the sack or by taking a sample at three points in the bag by means of a small hollow bag sampler about 8 inches long, with an opening 2 inches long near the point.

To show that these methods could not give a representative sample, two tests of graphite in sacks were made as shown in the table following and illustrated in figure 14.

Results of two tests of sampling sacked graphite.

[Figures and letters are same as in fig. 14.]

Position of sample.	Carbon.	
	Volatile.	Graphitic.
	Per cent.	*Per cent.*
T (1-5)	2.46	86.08
M (1-5)	1.78	87.32
B (1-5)	1.68	87.65
T (6-10)	2.25	84.74
M (6-10)	2.49	88.82
B (6-10)	2.10	87.63
1 (T, M, B)	2.56	88.93
3 (T, M, B)	2.26	84.90
5 (T, M, B)	2.67	83.71

In view of the considerations mentioned and the sampling results presented in the table, it is recommended that every bag be sampled continuously from top to bottom in the manner indicated in figure 14, by means of one of the devices described below.

One device consists of a tubular sleeve (fig. 15) 30 inches long and $\frac{3}{4}$ to $\frac{1}{2}$ inch in diameter, with a continuous slot extending from the point to within 4 inches of the top. Inserted in the sleeve is a rotating tube with a slot to correspond to the opening in the outer tube. Another suggested sampling device is a single tube with a check valve at the bottom for retaining the sample while the tube is being removed from the sack. It is further recommended that all samples be taken obliquely from top to bottom to get a fair proportion of the larger flakes which naturally slide farthest during the filling of the sack, and tend to collect at the sides.

The method of sampling shipments adopted by the Graphite Producers Association of Ashland, Ala., is an honest effort on the

part of Alabama operators to avoid disagreeable and costly disputes with the consumer.

As soon as a quantity of graphite is ready for shipment, the local chemist at Ashland divides the shipment into lots of 25 sacks. Samples are drawn from each bag and then combined to represent the individual lots. Analyses are made of the lot samples, and if any lot falls seriously below shipping grade, it is discarded from the shipment. The grade of the shipment is then calculated by averaging the analyses of the lot samples.

METHODS OF ANALYSIS.

The method of analyzing graphite products or ores has never been standardized to the extent of defining volatile and fixed carbon in a manner acceptable to all analysts. Accurate methods used in determining carbon in graphite products are tedious. Work recently

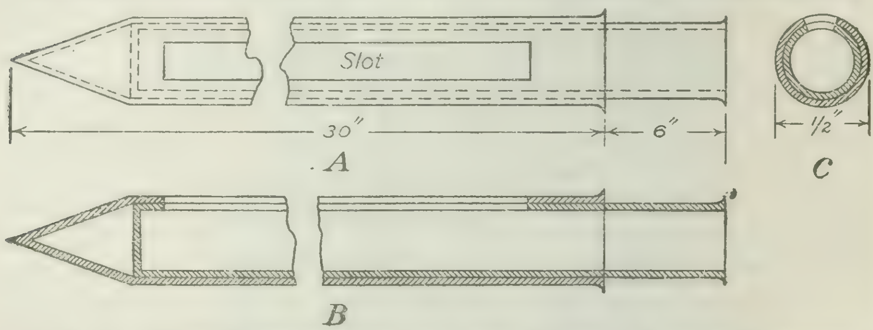


FIGURE 15.—Sections of sampling device (not drawn to scale). *A*, Longitudinal section, showing slot; *B*, longitudinal section, samples turned 90° from position *A*; *C*, cross section through slot.

done by the Pittsburgh experiment station of the Bureau of Mines on the chemical analysis of graphite products had in view the devising of a rapid, accurate, and simple method of analysis with a fair degree of accuracy. A description of this method is given on pages 43 to 45.

TENTATIVE SPECIFICATIONS.

In the past, crucible makers who used domestic flake graphite have as a rule bought this material only after examining samples submitted by producers. It may be helpful to suggest tentative specifications for No. 1 flake graphite.

The table following gives the screen and chemical analyses of graphite that is being used in the experimental work of the Columbus experiment station of the Bureau of Mines on graphite crucibles. All screen analyses were made at the Pittsburgh station of the bureau. A Tyler standard Ro-tap apparatus was used, and the graphite sample was screened for 45 minutes. All chemical analyses

were made at the Pittsburgh station, the volatile-carbon determinations of samples being made by heating for three minutes at 800° C.

Results of analyses of domestic flake graphite.

Sample No.	Kind of sample.	Carbon.		Cumulative percentages.				
		Volatile.	Graphitic.	Over 20 mesh.	Over 35 mesh.	Over 65 mesh.	Over 100 mesh.	Under 100 mesh.
		Per cent.	*Per cent.*					
1	Artificial graphite	1.27	87.82	0.3	11.7	54.9	95.9	4.1
2	No. 1 flake	1.68	82.02	....	.1	42.0	94.8	5.2
3	do	1.32	92.09	....	2.5	28.0	60.0	40.0
4	No. 1 flake and No. 2 flake	2.07	86.85	....	3.0	31.0	74.3	25.7
5	No. 1 flake	3.65	74.67	....	3.3	30.3	89.6	10.4
6	do	....	....	.1	15.4	69.6	95.6	4.4
7	Dust	6.06	41.33	....	.1	1.2	3.2	96.8
8	do	7.37	32.41	....	....	....	.2	99.8
9	do	7.20	31.47	....	....	.4	3.9	96.1
10	No. 2 flake	2.96	80.44	....	....	....	10.5	89.5
11	do	2.31	81.43	....	.2	9.9	67.6	32.4
12	No. 1 flake	....	....	....	5.7	58.5	93.0	7.0
13	do	1.98	86.18	.1	6.7	56.4	97.0	3.0
14	No. 2 flake	2.17	86.30	....	.1	.3	22.2	77.8
15	No. 1 flake	....	....	....	9.7	62.2	92.4	7.6
16	No. 1 flake, special	1.98	92.43	....	8.2	69.6	99.7	.3

Nine crucible manufacturers sent to the Bureau of Mines samples of graphite as prepared by them for incorporation in their crucible mixtures. Screen analyses of these samples were made at the Washington laboratory; the results showed considerable variation. The laboratory lacked the equipment usually found in establishments for the making of screen analyses, and consequently a nest of sieves of 20, 40, 60, 80, and 100 mesh had to be used rather than the standard meshing. For comparison, however, the results will answer just as well.

Results of analyses of manufacturer's samples at Washington laboratory.

No. of sample.	Manufacturer.	Cumulative percentages.						Volume of 100 grams (c.c).	Remarks.
		20 mesh.	40 mesh.	60 mesh.	80 mesh.	100 mesh.	100 mesh.		
1	A	1	68¼	83¼	92¼	94¼	100¼	106	Contains 35 per cent flake and 65 per cent Ceylon.
2	B	13½	57¾	71½	80¾	85½	99½	76	100 per cent Ceylon.
3	C	12½	88½	96½	98½	99	100	149	Madagascar flake.
4	C	16½	71	81½	89½	90	100	86	100 per cent Ceylon.
5	D	13½	62½	74¾	88	93½	99½	81	Contains 10 per cent flake and 90 per cent Ceylon.
6	E	23¼	66	76	83½	87	100	81	100 per cent Ceylon for steel "pot."
7	F	1¼	37¼	62¼	88¾	96¼	100	145	100 per cent Canadian.
8	G	14¼	58¼	75	85¼	89¼	99½	95	Contains 25 per cent flake; 75 per cent Ceylon for steel "pot."
9	H	4¼	49¼	69	80½	84¾	99¼	93	Contains 15 per cent flake and 85 per cent Ceylon.
10	I	40	74	84	89½	92	99½	89	100 per cent Ceylon.
11	I	16½	59½	75¾	86¾	90	99½	116	Contains 17 per cent flake and 83 per cent Ceylon.

CARBON CONTENT.

It is recommended that the graphitic carbon content of No. 1 flake be not less than 85 per cent. By graphitic carbon is tentatively meant the carbon remaining after the dried sample has been burned for three minutes at 800° C.

The table following gives the results of complete analysis of seven samples of crucible graphite. The analyses were made at the Pittsburgh station of the Bureau of Mines. The volatile carbon was determined by heating the samples at 800° C. for three minutes.

Results of complete analysis of seven samples of graphite.

Source of graphite	Alabama.	Alabama.	Alabama.	Alabama.	New York.	Pennsylvania.	Ceylon.
Sample No.	1	2	3	4	5	6	7
Volatile carbon	2.08	1.40	2.66	1.31	1.30	1.53	1.68
Graphitic carbon	84.52	90.58	81.82	91.18	88.97	88.80	85.06
SiO_2	7.02	3.99	8.33	4.08	4.34	5.24	7.81
Al_2O_3	5.06	2.96	6.35	2.31	2.40	2.05	2.82
Fe_2O_3	.53	.18	.39	.40	1.08	1.75	1.61
TiO_2	.14	.19	.13	.13	.38	.05	.13
CaO	....	....	....	....	.07	....	.19
MgO	.23	.16	.06	.08	.76	.09	.21
K_2O	.15	.23	.14	.31	.55	.08	.25
Na_2O	.008	.01	.11	.03	.12	.12	.11
SO_3	.007	.14	....	.01	....	.21	.005
P_2O_5	.06	....	....	.02	.02	.05	.05
MnO	....	....	....	....	....	.07	.04
CuO [a]	....	.14	....	....	....	....	....
ZnO [a]	....	....	....	....	....	....	.03
Total	99.805	99.98	99.99	99.86	99.99	100.04	99.995

[a] Contamination probably from brass screens.

The results presented indicate that the amount of deleterious impurities in domestic flake graphite is small. In the samples of Alabama graphite the Fe_2O_3 content did not exceed 0.53 per cent, and the combined content of alkalies and alkaline earths did not exceed 0.5 per cent. The amounts of mica and pyrite present were small indeed, judged from the alkali and SO_3 content. Even the Pennsylvania sample with a high Fe_2O_3 content showed only a small amount of SO_3, also a low alkali content. The sample from New York showed a combined alkali content of 1.5 per cent, and the Fe_2O_3 content was slightly more than 1 per cent.

If the single sample of Ceylon graphite analyzed may be taken as representative—and it should be stated that this sample was graphite prepared for use in crucibles—it is evident that, chemically, domestic graphite compares favorably with the Ceylon graphite.

SCREEN ANALYSIS.

The following tentative specifications as to screen analysis in cumulative percentages are recommended:

Recommended specifications for screen analysis.

[Figures represent cumulative percentages.]

Grade	No. 1 flake.
Over 35 standard mesh	35
Over 65 standard mesh	50
Over 100 standard mesh	100

Permissible allowance of not more than 3 per cent through 100 mesh.

PRESENT STATUS OF GRAPHITE MINING.

The request of the War Industries Board on August 10, 1918, that not less than 20 per cent of domestic flake graphite be incorporated into crucible-graphite mixtures during the balance of 1918, and 25 per cent thereafter, established a market for domestic flake which, it was hoped, would continue after the war. If domestic graphite can be used in this proportion without adversely affecting the quality of crucibles, it will be used provided it can be produced at costs that compare favorably with the prices paid for imported graphites. Under normal conditions the efficiency of crucibles is expressed as so many heats per crucible. However, during the war the vital comparison, as regards the national welfare, was the number of pounds of imported graphite used per heat. The introduction of every pound of domestic flake graphite that did not increase the consumption of imported graphite per crucible heat saved necessary ship tonnage. The proportion of domestic flake that reduces the consumption of imported graphite to a minimum has not yet been determined.

In all of the graphite mining districts in this country operating costs are very high, and in normal times, if imports were unrestricted, the domestic mining industry could not compete with the industries in Madagascar and Ceylon. On both of these islands, the deposits are of large extent and of such high grade that a simple cobbing or washing represents the only milling necessary. Labor is much cheaper in the Far East than in the United States. Therefore, in spite of the great distance from the market, graphite from these islands could be placed on the docks in this country at prices which at present would provide no profit for domestic producers. Also, overproduction abroad might tend to force prices still lower through dumping of surplus production on the American market.

Operating costs in the United States in October, 1918, ranged from 6 to 14 cents, with an average of 10 cents, per pound of No. 1 flake. No allowance has been made for depletion and depreciation, which would add 1 to 2 cents per pound to the costs stated. In arriving at these costs the production of No. 1 flake has been charged with all operating expenses and has been credited with the miscellaneous income derived from the sale of No. 2 flake and dust. The wide

variation in costs is due to differences in the efficiency of individual plants as well as to fluctuations in the grade of ore treated by the different plants.

The graphite in the Alabama district is of lower grade than that in any of the other districts mentioned in this report. With an average recovery of 19 pounds of No. 1 flake per ton of ore and an after-war price of 9 cents per pound, which is 3 to 4 cents higher than the pre-war price, it is evident that, even though all the ore is mined in open pits, only those plants efficiently managed and working comparatively rich deposits will survive unless new and important uses develop for the by-product grades so as to warrant the establishment of a local central factory for producing commercial graphite products. Many of the Alabama plants are 6 to 9 miles from the railroad and are in localities where most of the roads are difficult to maintain because they are of clay and poorly drained. At plants so situated the cost of transportation becomes a serious factor in operating expenses.

Until 1913, the graphite production in Alabama was small, but has steadily increased so that since 1915 Alabama has been the leading graphite-producing State in the Union.

Milling capacity in the Alabama district, with the 39 plants now in operation or building, is sufficient to produce a large quantity of graphite, if these mills could all run 20 hours a day. On the assumption that each mill would average 3,000 pounds of No. 1 flake daily, which is a conservative estimate, a total production of nearly 60 tons a day could be obtained. The actual production of No. 1 flake from this district during 1918 averaged less than 350 tons per month.

In New York and intermittently in Pennsylvania, graphite was mined before the war. New York will undoubtedly continue to produce graphite after the war in somewhat greater quantities than before. Both of these districts are close to a large market. The recovery of graphite per ton of ore is double that obtained in Alabama; this is a distinct advantage, particularly when by-product grades can be sold advantageously.

The costs of production are practically the same as those in Alabama, chiefly because most of the plants in New York and Pennsylvania have just started producing; but with reversion to peace times, these districts have a distinct advantage over the Southeastern district, in spite of the necessity in New York for underground mining.

In the latter part of 1918 the New York district was producing not more than 90 tons of graphite per month, of which probably 60 per cent would be No. 1 flake. The Pennsylvania field produced about 50 tons of graphite per month, of which about 60 per cent would be No. 1 flake.

The Texas district, if provided with an outlet for by-product grades of flake graphite, would have a good opportunity of surviving post-war readjustments. The production from this district has not been large, but could be considerably increased without great difficulty. With respect to a Western and Central market, the Texas deposits are favorably situated. The deposits are not only of large extent but are high grade as well.

These statements have been made in order to indicate, in a general way, the precarious position of the mining of domestic crystalline flake graphite. It would be presumptuous to suggest methods by means of which the mining of domestic graphite can be established firmly on a postwar basis, for underlying the entire consideration is the fundamental economic law of supply and demand. As regards domestic flake graphite, the supply exists and can be obtained. If the demand can be created or extended by scientific, rather than by artificial means, the supply will be forthcoming whenever the price is high enough to justify production.

With the resumption of peacetime activities, the demand for crucibles and other graphite products has slackened. This, of course, has reacted upon the domestic graphite mining industry, with the result that production has been greatly curtailed. Until business in the United States has resumed its normal peace-time trend, some method of control or regulation will be necessary to prevent the collapse of certain industries, which the lessons of the present war have taught should not be permitted to die. To be independent of foreign graphite, the use of 100 per cent domestic flake in crucibles will have to be developed, if the graphite crucible continues to be used as a medium for melting alloys of all kinds. It is possible, of course, that the use of some refractory other than graphite, or the extended use of melting furnaces requiring no crucibles may develop to such a point as to make present practice obsolete.

METHOD FOR RAPID ANALYSIS OF GRAPHITE USED BY THE BUREAU OF MINES.

By G. B. Taylor and W. A. Selvig.

In the following paragraphs are outlined certain methods of graphite analysis used by the Pittsburgh laboratory of the bureau. These are intended to provide a simple and rapid procedure for the proximate analysis of graphite, such as can be performed in the average laboratory in the minimum of time, together with a fair degree of accuracy. The proximate analysis consists of the determination of moisture, volatile matter, ash, and graphitic carbon by difference.

PREPARATION OF SAMPLE.

On account of the difficulty of grinding flake graphite, no degree of fineness can be fixed; however, for material containing considerable gangue, grinding to pass through a 60-mesh sieve is recommended. The flakes of graphite separated during the screening must of course be included in the 60-mesh material, and must be thoroughly mixed before portions for analysis are weighed out.

DETERMINATION.

MOISTURE.

One gram of the sample is placed in a weighed porcelain capsule $\frac{5}{8}$-inch deep by $1\frac{3}{4}$ inches wide, and heated for one hour at 105° C. in a constant-temperature oven. The capsule is then removed from the oven, covered, and cooled in a desiccator over sulphuric acid. The loss in weight multiplied by 100 is recorded as the percentage of moisture. If platinum crucibles are available, it is preferable to use a 25-c.c. platinum crucible instead of the porcelain capsule.

VOLATILE MATTER.

The covered capsule containing the graphite from the moisture determination is placed in a muffle furnace and heated at 800° C. for three minutes, removed from the furnace, cooled in a desiccator, and weighed. The loss in weight multiplied by 100 is recorded as the percentage of volatile matter.

A lower temperature and a shorter time interval than is used in the standard method for the determination of volatile matter in coal is recommended, as the volatile-matter content in graphite is small, and the oxidation and loss of graphitic carbon should be cut down to a minimum.

ASH.

Ash is determined in the residue from the volatile-matter determination. The capsule containing the residue from that determination is placed in a muffle furnace at about 800° C. and is heated until completely burned. The capsule with its contents is removed from the muffle and cooled and weighed, after which it is replaced in the muffle, heated for half an hour, cooled in a desiccator, and weighed again. This heating and weighing should be continued until the change in weights between two consecutive ignitions is 0.0005 gram or less.

The weight of the crucible plus the ash, minus the weight of the empty crucible, multiplied by 100 is recorded as the percentage of ash.

GRAPHITIC CARBON.

Graphitic carbon, which is determined by calculation, is the difference obtained by subtracting the sum of the percentages of moisture, ash, and volatile matter from 100.

GRAPHITIC CARBON BY COMBUSTION.

The most accurate method of determining graphitic carbon is by combustion, and subsequent weighing of the CO_2 formed. While it has been found that graphitic carbon, as calculated, checks very closely with the graphitic carbon determined by direct combustion, the latter should be used when greater accuracy is desired. This method, however, calls for special equipment and requires considerable skill in manipulation.

On account of the possible presence of carbonates in ore, and possibly small amounts of flotation oils in the sample, if a concentrate, a preliminary treatment before combustion is required.

PRELIMINARY TREATMENT OF ORES.

A sample, weighing 0.2000 to 1.0000 gram, depending on the relative amount of graphitic carbon, is placed in a 100-c.c. evaporating dish with 25 c.c. of 1 to 1 HCl, heated over a hot plate for 15 minutes, filtered through a filter of ignited asbestos, and washed with hot water until free from chlorides. The filter and the residue are then transferred to a porcelain or platinum boat and dried on the hot plate. The boat containing the residue is transferred to the tube of a combustion furnace and burned in a stream of oxygen gas, the CO_2 formed being collected in a potash bulb with a 30 per cent KOH solution. The combustion tube should contain some fused lead chromate to retain any sulphur that may be present.

PRELIMINARY TREATMENT OF CONCENTRATES.

Concentrates are likely to contain flotation oils, which must, of course, be removed before combustion, or too high results will be obtained for graphitic carbon.

This may be done by placing a sample weighing 0.2000 to 0.5000 gram in a small Erlenmeyer flask and adding about 25 c. c. of ether, corking loosely, and allowing to stand for about one-half hour, shaking at intervals. The mixture is filtered onto asbestos in a Gootch crucible, the asbestos having previously been washed with hydrochloric acid and ignited. The residue on the filter is washed with alcohol and next with distilled water; it is then ready to be treated with acid for the removal of possible carbonates, as is done in the treatment of ores.

REFINING ALABAMA FLAKE GRAPHITE FOR CRUCIBLE USE.

By Frederick G. Moses.

INTRODUCTION.

In order to determine the possibility of producing a satisfactory crucible graphite from Alabama flake graphite, an investigation embracing a large number of tests was made at the Salt Lake City station of the Bureau of Mines. The following report contains the information obtained during the progress of this investigation.

A primary object of the work was to ascertain the most satisfactory means by which the low-grade graphite concentrates produced from Alabama ores could be made into satisfactory crucible stock. Of course, the success of any one of the methods of treatment developed will depend both on the amount of satisfactory material that can be recovered by the finishing process, and on the ease and cheapness of the operation itself. In all of the experiments both of these factors were kept in mind.

REQUIREMENTS FOR CRUCIBLE GRAPHITE.

After an extended investigation into the specifications for graphite satisfactory for use in crucible manufacture, Geo. D. Dub, has suggested the following specifications for satisfactory No. 1, Crucible Flake[a]. For the sake of emphasis they are summarized here.

Dub suggested that a No. 1 flake is satisfactory, as regards carbon content when the content of graphitic carbon is not less than 85 per cent. Graphitic carbon is here intended to mean that carbon which remains after burning a dried sample three minutes at 800° C.

It is also suggested that 35 per cent of the product should remain on a 35-mesh standard screen, 30 per cent should pass through a 35-mesh and remain on a 65-mesh standard screen, and the remaining 35 per cent should be between 65 and 100 mesh. An allowance of 3 per cent through 100 mesh could be permitted.

The aim of the investigation herein reported was to produce material of the grade outlined from the concentrates of graphite from the Alabama graphite fields.

PRESENT PRACTICE.

The production of crucible stock from the Alabama ores involves two distinct steps. The first is a concentration process by which a

[a] See page 40 of this bulletin for "carbon content" and page 39 for "screen analysis."

comparatively low-grade concentrate is recovered from the ore. The second step comprises the conversion of this low-grade concentrate into a high-grade material that will be satisfactory for crucible making.

The first step, being simply concentration, need not be discussed here. The second step, finishing, as it is called, is much more difficult to carry out and is the one that demands the greatest amount of investigation and improvement.

The present method of finishing the crude concentrate consists of, drying the concentrate, grinding the dried material in a buhr mill, and "bolting" or screening the ground product to remove the impurities that have been crushed during the grinding. The theory is that the graphite particles, being tougher, smoother, and thinner than the impurities, will be rubbed and polished between the buhrstones with a minimum reduction in size, while the impurities themselves, owing to their more brittle characteristics and more massive structure, will be ground fine enough for removal by screening over a suitable cloth.

The method is simple, and in fact, within certain narrow limits and under favorable circumstances, gives good results. As a general rule, however, two difficulties are encountered that interfere with its commercial success—the grinding often destroys a large proportion of the coarse and valuable flake and there is a lack of uniformity in the finished product. The primary object of the present investigation was to determine the most efficient manner of obtaining the desired results and eliminating the troubles in the method mentioned above.

CONCENTRATES USED IN TESTS.

As has been suggested, the problem involved is the treatment of the concentrates that are being produced in the Alabama district. Therefore, all of the work done during this investigation was on a series of representative samples from that district.

The concentrates produced in the Alabama district differ widely in their physical characteristics and graphitic content. However, as the physical properties of the different minerals composing the concentrates can not vary to any considerable extent, a scheme worked out for any one concentrate can be applied, with only slight variations and adjustments, to the other concentrates. For this reason, and because of the numerous schemes tried in the limited time available for the work, it was impossible to conduct tests of all of the concentrates. Therefore, it was decided to select several of the most representative for investigation, and to determine the extent that the results obtained on these concentrates could be generalized and applied to all.

Table 1 gives the results of screen analyses and assays of the different samples selected.

TABLE 1.—*Results of screen analyses and assays of different samples selected for experiment.*

Mesh.	Percentage by weight.	Carbon assay (per cent).	Units of carbon, each size.[a]	Percentage of total carbon.
Sample 1:				
14	0.1			
20	1.1	74.8	82.0	1.82
28	5.4	60.8	328—	7.27
35	14.8	53.6	793.0	17.60
48	22.5	47.2	1,062.0	23.55
65	21.8	44.4	968.0	21.47
100	17.4	44.00	766.0	16.99
150	8.8	43.40	382.0	8.47
200	2.7	41.20	111.0	2.46
−200	4.5	3.60	16.0	.35
Total			4,508.0	99.98
Sample 2:				
14	1.8	6.1	11.0	.21
20	2.4	15.7	37.7	.74
28	5.1	38.3	195.4	3.81
35	10.2	51.9	529.0	10.32
48	20.6	56.7	1,169.5	22.81
65	22.6	58.2	1,313.0	25.61
100	24.7	55.2	1,361.5	26.55
150	8.8	46.7	407.0	7.94
200	1.4	46.8	65.7	1.28
−200	2.3	16.2	37.2	.73
Total		51.27	5,127.0	100.00
Sample 4:				
14	Tr.			
20	Tr.			
28	.5	24.0	12.0	.37
35	6.2	20.7	128.0	3.92
48	16.1	25.9	417.0	12.79
65	23.1	33.1	765.0	23.46
100	25.7	43.4	1,114.0	34.16
150	16.1	49.1	780.0	23.92
200	4.0	56.1	22.0	.67
−200	6.5	36.9	23.0	.71
Total			3,261.0	100.00
Sample 5:				
14	.4			
20	1.8			
28	5.5	52.8	290.4	4.26
35	14.3	69.6	995.3	14.60
48	25.7	72.4	1,860.7	27.29
65	26.3	73.4	1,930.3	28.32
100	21.0	67.4	1,415.4	20.76
150	4.5	64.8	291.5	4.29
200	.3			
−200	.9	36.0	32.4	.48
Total			6,816.0	100.00
Sample 6:				
14				
20	.4			
28	2.1	60.2	126.42	2.98
35	7.7	53.6	412.72	39.82
48	16.4	48.2	790.48	18.27
65	22.4	45.2	1,012.48	23.82
100	24.5	35.6	872.20	20.55
150	17.0	38.4	652.80	15.42
200	4.0	39.0	156.00	3.68
−200	5.5	40.4	222.20	5.35
Total			4,245.30	99.89

[a] Column 2 × column 3.

The characteristics of the samples were briefly as follows:

No. 1. A fairly coarse concentrate assaying 48.95 per cent carbon.

No. 2. A coarse flake concentrate assaying 56.47 per cent carbon.

No. 4. A fine flake concentrate assaying 42.38 per cent carbon.

No. 5. A medium coarse flake concentrate assaying 73.30 per cent carbon.

No. 8. A medium coarse flake concentrate assaying 42.45 per cent carbon.

As the work progressed, it was found that duplication of all of the necessary experiments on even this number of samples would involve too much time; hence two of the most representative were selected for investigation, and the conclusions drawn from the results obtained from them were applied to the rest. The two samples chosen for the work were Nos. 2 and 4, as they represented two general classes of concentrate—a fine and a coarse medium-grade material.

PHYSICAL PROPERTIES OF GRAPHITE AND ASSOCIATED GANGUE MINERALS.

It is evident that production from the concentrates described of a graphite stock containing 85 per cent or more carbon would first involve separation of the graphite from its contained impurities. This can be accomplished only by taking advantage of differences between the physical properties of the graphite and of the associated impurities. For comparison of the physical properties that might be utilized for this purpose they have been tabulated in Table 2, as follows:

TABLE 2.—*Physical properties of graphite and of associated minerals.*

Mineral.	Specific gravity.	Hardness.	Brittleness.	Shape.	Color.	Electroconductivity.	Surface condition.	Cleavage.
Graphite	2.1	1 to 2	Not brittle	Flat, tabular plates.	Dark gray and black.	Good	Metallic luster.	One direction.
Quartz	2.66	7	Very brittle	Massive, granular, or compact.	Colorless, or may be any color.	Nonconductor.	Glassy or vitreous.	Practically absent.
Mica	2.8	2 to 2½	Not brittle	Thin, scaly plates.	Colorless, or pale green to brown to black.	do	Smooth, glassy, or vitreous.	Nearly perfect in one direction.

Some of these properties vary greatly, but others are closely alike. Unfortunately the property of specific gravity, most often used in the separation of minerals, can not be used in finishing graphite because the weights of the various substances involved are nearly the same. On the other hand, properties that are the least often used in the commercial separation of minerals, for instance, electrical conductivity, are the properties that show the greatest amount of difference and must be depended upon to give the best results.

Thus the standard equipment manufactured for the separation of minerals, such as gravity tables, can not be used to the best advantage in finishing graphite. In fact, past experimental work with specific

gravity machines has substantiated this conclusion. Also, because no satisfactory equipment has been developed to make a separation by taking advantage of the little used physical characteristics, it was necessary to construct special apparatus.

ASPIRATOR TESTS.

Each apparatus investigated during this work uses the physical properties or characteristics of the graphite and gangue minerals as a means to accomplish separation.

The first experiments were made to determine the possibility of separating the graphite and gangue material by differences in specific gravity and shape of the various particles. The apparatus first tried depends on the difference in specific gravity and the difference in shape of the various minerals to accomplish separation. This type of apparatus is already being used to a limited extent in Alabama and is there known as an "aspirator."

The aspirator is fundamentally an air classifier. This name is appropriate for the reason that the separation depends on the settling rate of the different minerals in a current of air.

The apparatus (Pl. I, A) constructed for the experiments was closely similar to those that are being used in the Alabama field at the present time. The material to be treated is fed from a hopper to a roller from which it drops into the large end of a long, sloping, horizontally placed box. The roll feeder is arranged so as to feed the material in an even curtain across the whole opening. From the small end of the box a pipe leads to a suction fan. The box is 13.5 inches wide by 12.5 inches high at the feed end, and 43.5 inches long. The suction pipe is 31 inches long and 4 inches in diameter.

Across the opening of the box and parallel to the ground line are four vanes or sand deflectors, placed at such an angle from the vertical that any material fed from the roller when the air current is off will be deflected to the outside of the box.

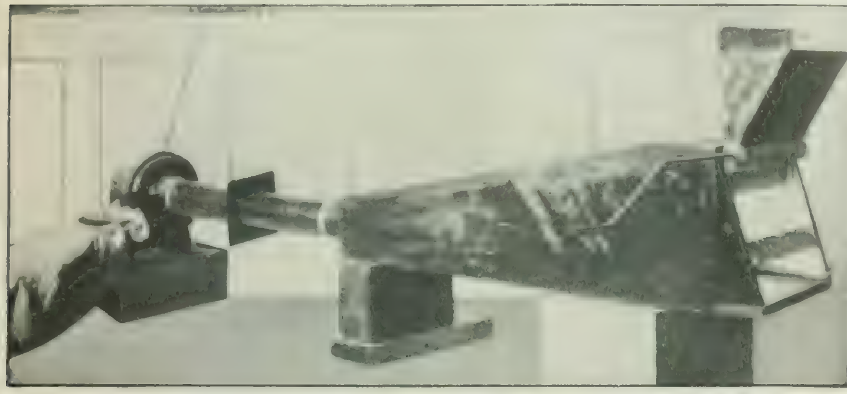
For a description of the operation of the classifier see page 25.

Several samples of the concentrates already described were tested with this machine, representative results of several runs being as follows:

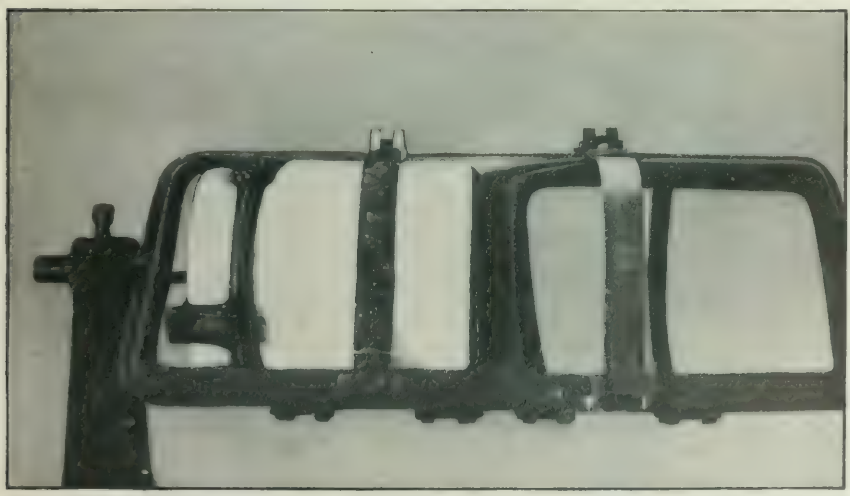
	Graphite, per cent.
Heads	51.23
Concentrates	69.23
Tailings	19.08

These results show a calculated recovery of 86.25 per cent of the total graphite in the concentrates produced.

The aspirator yields three products, as follows: Sand tailings, which are recovered on the outside of the box below the feed hopper; coarse graphite concentrates, which are heavy enough to be carried into the box and are deposited there, and the pure, fine concentrate



A. IMPROVISED ASPIRATOR USED IN TESTS AT SALT LAKE CITY STATION.



B. BUHR MILL USED IN GRAPHITE GRINDING TESTS.

that is too light to be deposited within the box, but is sucked through the blower and caught in a bag at its outlet.

In the particular test described. 87.1 per cent of the concentrate was deposited in the box and the remaining 12.9 per cent was drawn through the fan. The former material assayed 68.4 per cent graphite: the fine material assayed 74.85 per cent.

In order that the difference in the sizes of the various products made may be compared with the material before treatment, and with each other, the results of screen analysis and of assay of each of the sizes are given in Table 3 following. In this table the results for fine and coarse concentrates were combined.

TABLE 3.—*Results of screen analyses and of assays of aspirator concentrates.*

COARSE CONCENTRATES.

[87.1 per cent of aspirator concentrates.]

Mesh.	Proportion by weight.	Carbon assay.	Carbon units.[a]	Total carbon.
	Per cent.	*Per cent.*		*Per cent.*
14	Tr.			
20	0.4	83.7	33.5	0.49
28	2.6	89.0	231.8	3.39
35	10.6	87.4	926.5	13.55
48	24.4	80.8	1,972.0	28.83
65	25.4	69.0	1,753.0	25.63
100	28.3	62.0	1,756.0	25.68
150	7.1	24.4	173.2	2.53
200	.5	23.3	11.5	.17
−200	.5	23.4	11.5	.17
Total	99.8	68.39	6,839.0	100.44

[a] Column 2 times column 3.

FINE CONCENTRATES.

[12.9 per cent of aspirator concentrates.]

14	None.			
20	None.			
28	Tr.			
35	1.6	88.1	141.00	1.70
48	9.2	90.3	831.00	10.92
65	24.1	87.8	2,118.00	28.11
100	37.8	81.1	3,063.00	40.74
150	17.1	62.8	1,073.00	14.17
200	3.9	48.8	190.90	2.36
−200	5.8	29.2	169.00	2.08
Total	99.5	74.85	7,585.00	100.08

ASPIRATOR TAILINGS.

14	5.00	4.1	20.5	1.05
20	8.00	12.9	103.2	5.41
28	12.80	23.2	297.5	15.59
35	18.50	26.9	498.0	26.10
48	28.40	24.0	681.5	35.69
65	20.00	13.2	264.0	13.83
100	6.40	5.9	37.8	1.98
150	.20	12.3	2.5	.13
200	Tr.			
−200	.20	15.3	3.0	.15
Total	99.5	19.08	1,908.0	99.99

It should be noted that the greatest concentration and improvement of product is in the coarser material of the coarse concentrates. The great difference between the carbon content of the material coarser than 150 mesh and of that finer than this size is very striking and also important.

As regards the fine concentrates produced the large percentage (83.02), which is −48 mesh and +150 mesh, is noteworthy as is the high carbon content of the +200 material.

Treatment of another sample, No. 4, in the aspirator gave the following results, showing a calculated recovery of 96.2 per cent of the total graphite in the concentrates produced:

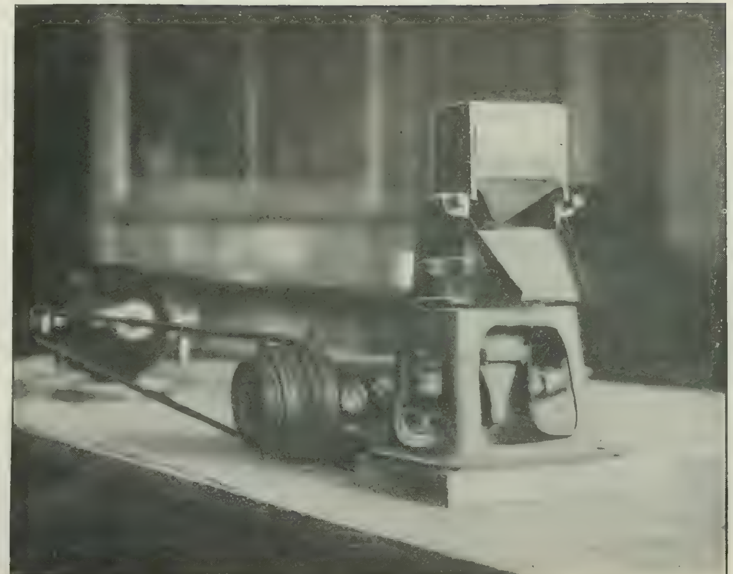
	Carbon, per cent.
Heads	42.61
Concentrates	56.62
Tailings	6.03

These results indicate that although some of the impurities can be removed with the aspirator, thereby materially raising the grade of the material, the application of this type of aspirator will never be general.

It has long been known that the coarse sand particles cause the greatest destruction of the large flake in the buhr mill. When the concentrates carry a large proportion of sand particles of such a nature that the type of treatment described will raise the grade of the crude product to 70 per cent or better, the aspirator can undoubtedly be used to great advantage before treating the concentrates in the buhr mill. However, unless there is a comparatively large proportion of such impurities in the crude concentrate, the aspirator can not be used to the best advantage. The apparatus should certainly not be incorporated in a finishing mill except after thorough and comprehensive tests to determine the results that can be obtained by its use, and then only on the advice of a competent engineer conversant with the conditions that must be met by the installation.

Probably the most potent reason for the comparatively poor results obtained with the aspirator was the wide variety in size of the material treated. When a current of air strong enough to remove the coarse graphite flakes from the feed was used, the fine sand would, at the same time, be drawn into the box with the concentrate.

However, when the concentrates are of low grade and contain a large quantity of coarse sand, treatment by such a simple process as the one just described is so advantageous that its use should be considered, and when feasible, the designing engineer should include an aspirator in a projected plant.



A. PNEUMATIC JIG USED IN TESTS.

B. BUHR MILL USED IN TESTS.

TESTS WITH PNEUMATIC JIG.

The pneumatic jig was the next apparatus tested. Its construction differs greatly from that of the aspirator, although the principles involved in the operation of each are similar. As this equipment has been manufactured on a commercial scale for several years, its use would be advantageous when feasible, as a more simple and standard "layout" could be arranged than where improvised equipment like that described was used. Both types would be simple to operate, the aspirator being capable of more sensitive adjustment.

The jig used in the graphite tests is a small laboratory apparatus, built by the manufacturers of the standard machine, for laboratory experiments. Plate II, *A*, shows the jig as set up for work. It comprises a small metal box, the bottom of which is made of a screen 3.25 by 2.5 inches. Air pressure of 2 to 4 ounces, furnished by a pressure blower or compressor is admitted to the bottom of the box under the screen through a small rapidly rotating valve. The rapid pulsation of the air, due to its passage through the valve, causes the material on the screen to remain in continuous agitation and partial suspension. Consequently, the coarse sandy impurities, being heavier, collect at the bottom, the lighter graphitic material being stratified above the sand. The jig is so arranged that operation is continuous, the sand being constantly collected and discharged from one side, while the lighter graphite is separated above the sand and discharged from the top of the column and on the other side.

Several tests were made with the jig, representative results being as follows:

Results of two representative tests with pneumatic jig.

	Carbon, per cent— Sample 2.[a]	Sample 4.[b]
Heads	51.23	42.61
Concentrates	69.66	64.13
Tailings	15.11	3.14

It is seen that the recovery of clean tailings from the treatment of sample 4 was higher than from the treatment of sample 2. In order that the products after treatment can be compared with the original feed as to size and carbon content, the screen analysis and related data are given in Table 4 following:

[a] A calculated recovery of 91.25 per cent of the graphite in the jig concentrate is indicated.

[b] A calculated recovery of 97.5 per cent of the graphite in the jig concentrate is indicated.

TABLE 4.—*Results of screen analyses and of assays of jig concentrate and tailing.*

[Sample 1.]

Mesh.	Concentrate.				Tailing.			
	Proportion by weight.	Carbon assay.	Carbon, units.[a]	Total carbon.	Proportion by weight.	Carbon assay.	Carbon, units.[a]	Total carbon.
	Per cent.	Per cent.		Per cent.	Per cent.	Per cent.		Per cent.
14	None.				0.1	5.2		
20	Tr.				.4		2.1	0.66
28	0.4	80.9	32.5	0.51	2.6	5.7	14.8	4.63
35	3.1	78.4	242.5	3.78	15.7	4.0	62.8	19.65
48	10.5	76.4	802.5	12.51	28.6	3.4	97.2	30.42
65	20.8	69.7	1,450	22.61	27.8	3.1	86.2	26.98
100	29.1	64.5	1,875	29.23	18.7	2.6	48.6	15.21
150	21.1	62.6	1,322	20.61	9.8	.8	7.8	2.44
200	5.8	55.1	319.5	4.98	.2			
—200	9.3	39.8	370	5.77	.3			
Total	100.1	64.12	6,414	100.00	99.2	3.19		

[a] "Proportion by weight" times "carbon assay."

The results given in the table indicate that the air jig has practically the same field of application as the aspirator, also that it can be made to produce a cleaner tailing than the aspirator, but the aspirator will give a higher grade of finished product. This condition will not, however, interfere with the jig's usefulness, provided the material it removes from the crude concentrate is the coarse sand that causes the high loss of coarse flake in the buhr mill. An inspection of the various products of the jig will indicate that it does remove the coarse sand particles.

Many of the remarks regarding the use of the aspirator apply also to the air jig. There is no question that under certain circumstances the jig can be made to do valuable work in the preparation of crude graphite for crucible making, but the character of the crude concentrates themselves, and the other factors that enter into the commercial finishing of concentrates must be considered before the jig be recommended for use in preliminary treatment. If the jig is used only to do that work for which it is particularly adapted, the removal of coarse sand impurities, its success is assured.

TESTS WITH PEBBLE MILL.

Probably the most striking characteristics of graphite flakes are their toughness and shape. In these two respects the flakes differ greatly from the impurities usually associated with the graphite in the crude concentrates. It was hoped, therefore, that these properties could be utilized in separating the graphite from the associated impurities.

It was thought that the coarse sand impurities, although much harder than the graphite flakes, were so much more brittle that the concentrate could be so ground as to reduce materially the size of the sand particles without causing a material reduction in the size of the graphite particles. A careful survey of the different classes of grinding machinery indicated that this operation could be carried out with greatest chance of success in a mill of the ball or pebble type.

The difference in the specific gravities of the two materials was also a reason for believing that this sort of selective grinding could be done. It was thought that under the correct conditions of dilution the heavier sandy material would tend to classify in the bottom of the charge among the grinding balls where it would be ground more than the graphite, which, because of its lighter weight would tend to segregate higher in the charge and out of the grinding zone. Such a separation corresponds closely to that in a hydraulic classifier. The results obtained from experiments proved that in most particulars the assumptions were fundamentally correct.

After a treatment such as suggested, which would produce a product with the sandy constituents ground finer than the flake graphite, two methods of separating the sand and graphite are available.

One of the methods is by screening. Experiment showed that after such grinding, the grade of the original material could be raised 25 per cent or more by simply passing the ground concentrate over a 100-mesh screen. It will be recalled that 100-mesh material is the smallest size that can be satisfactorily used in crucible making.

The other method is by flotation, the process by which much of the crude concentrate produced in the Alabama field is made. Tests revealed that in order to recover the coarse flake in the ore by flotation, it was necessary to make a fairly low grade concentrate. The reason seemed to be that some of the silicious material was porous and sponge-like in structure, and contained quantities of entrained air which invariably caused the sand to float with the carbon.

Wet grinding of the concentrate in a ball mill tended to break up the pumice-like silica and to eliminate the entrapped air. When this had been done, the silica did not float as readily as before. Therefore, after grinding, the concentrate can be refloated and its grade raised to a degree corresponding to the amount of the spongy sand removed.

A small laboratory pebble mill (see Pl. I, *B*, p. 50) was used in the grinding tests. The charges consisted of 50 per cent water and 50 per cent crude concentrate. The grinding periods varied, but at first were of 30 minutes' duration.

A certain sample contained 90 per cent of the carbon as material coarser than 100 mesh which assayed 53.9 per cent carbon before grinding. After grinding in the pebble mill, 81 per cent of the carbon still remained on a 100-mesh screen as material which assayed

61.3 per cent carbon. These results indicated that a decided improvement had been made in the grade of the material coarser than 100 mesh, with a loss of only 9 per cent of the valuable coarse flake.

On the strength of these results, it was decided to make a series of tests under the conditions already described, but varying the length of the grinding period in each test. The results of this series of experiments are given in Table 5 following:

TABLE 5.—*Results of grinding graphite samples in pebble mill with equal parts of water.*

	Time of grinding (hours).	Grade of concentrate (percentage of carbon in +100-mesh material).	Carbon remaining on 100-mesh screen.	
			Percentage of total carbon of original sample.	Percentage of +100-mesh carbon in original sample.
Sample 2	0	53.9	90.00	100.00
	½	61.3	81.00	90.90
	¾	77.6	80.60	89.60
	1	81.7	83.30	92.50
	1¼	79.8	81.40	90.40
	1½	81.4	72.60	80.60
	2	82.1	67.40	74.80
Sample 4	0	47.9	80.5	100.00
	½	70.6	65.9	81.80
	¾	74.6	62.7	77.90
	1	81.4	62.5	77.60
	1¼	75.4	58.7	72.90
	1½	80.5	59.3	73.60
	2	82.4	51.1	63.50

The results are plotted in figures 16 and 17. The figures show that the correct time for grinding, under the conditions of this set

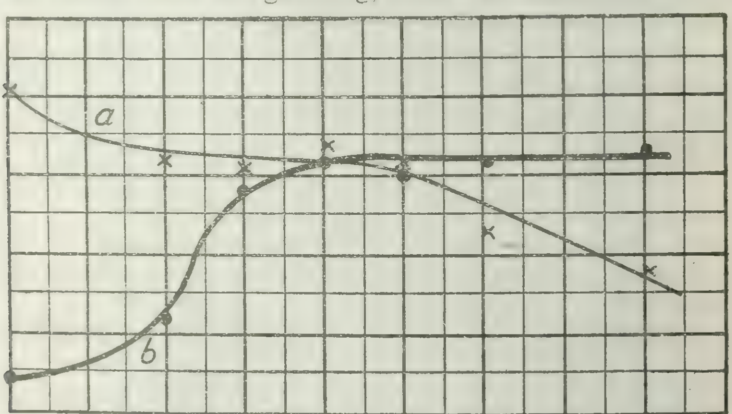


FIGURE 16.—Curves showing results of grinding sample 2 in pebble mill with equal parts of water. *a*, Recovery; *b*, carbon

of experiments, is the time that will produce the highest grade of +100-mesh material with a minimum destruction of the flake that originally existed in the concentrates coarser than 100 mesh.

On the curve this point will be at the intersection of the line representing the assay of the +100-mesh material with the line representing the recovery of +100-mesh carbon. For sample 2 the time is about one hour, whereas for sample 4 it is about 30 minutes.

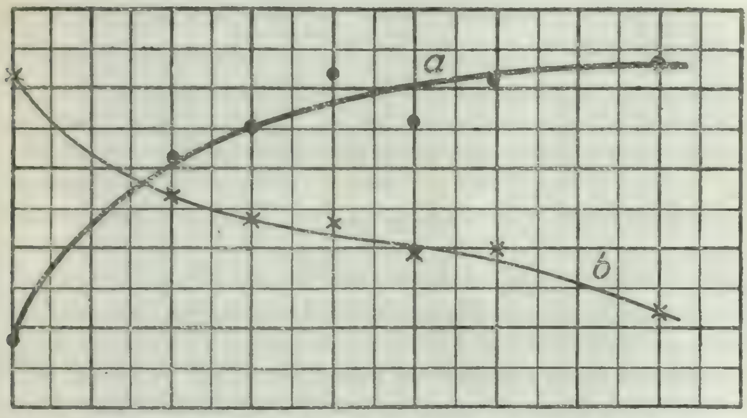


FIGURE 17.—Curves showing results of grinding sample 4 in pebble mill with equal parts of water. *a*, Carbon; *b*, recovery.

It is evident from these results that the conditions that give the best results will vary with the physical characteristics of the crude concentrate itself, and must be determined by actual experiment with the material that is to be treated in the commercial plant.

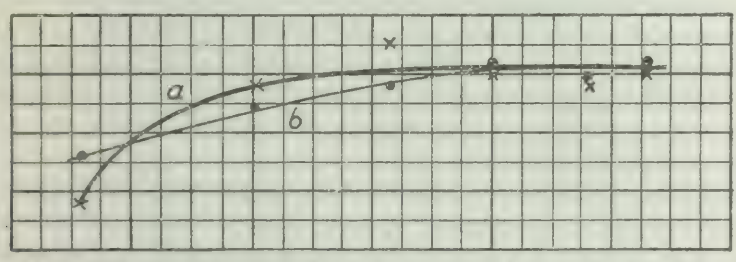


FIGURE 18.—Curves showing results of grinding sample 2 in a pebble mill with different percentages of water. *a*, Recovery; *b*, carbon.

In order to determine the effect of varying the amount of water in the pebble-mill charge, several tests were made, with sample 2, the other conditions being similar to those that had been proved to be the most satisfactory in the previous series. The results of these tests are shown in Table 6 following and in figure 18. The concentrates were fed to the mill at the rate of 400 grams per hour.

TABLE 6.—*Results of grinding graphite sample 2 in pebble mill with different percentages of water.*[a]

Quantity of water used (c.c.)	Grade of concentrate (percentage of carbon in +100-mesh material).	Carbon remaining on 100-mesh screen.		Water in mill pulp (per cent).
		Percentage of total carbon of original sample.	Percentage of +100-mesh carbon of original sample.	
0	53.9	90.00	100.00	0.0
300	77.6	73.7	81.90	42.8
400	81.7	83.3	92.50	50.0
500	83.5	87.0	96.60	55.55
600	85.0	84.50	93.30	60.0
700	84.1	83.40	92.50	63.6
800	84.7	85.10	94.50	66.6

[a] See also figure 18.

Figure 18 shows that no improvements in the results are to be noted after a dilution of 60 per cent water is reached. In this experiment the grade of the +100-mesh material was raised from 53.9 per cent carbon to 85 per cent carbon, with a loss of only 7 per cent of the carbon that was coarser than 100 mesh before grinding.

DISCUSSION OF PEBBLE-MILL RESULTS.

In summarizing the results obtained by the pebble-mill grinding, it may be said that the apparatus, when operated under the conditions that have been determined to be the most satisfactory, will raise the grade of the +100-mesh material in a crude concentrate to such a degree that the following buhr-mill grinding can be carried on to give the maximum results possible with it. At the same time, the results indicate that the pebble mill can not be used to produce a finished grade of product. In other words, the pebble-mill grinding should be used only as a step in the process, and preparatory to treatment in the buhr mill.

The choice of method of removing the impurities that are ground in the pebble mill will depend on the character of the concentrates that are being treated and other conditions that will be encountered in the commercial finishing plant.

The results obtained in the experiments here described indicate clearly that pebble-mill grinding has a wide field and can be made to produce favorable results even under the most adverse and difficult operating conditions.

TESTS WITH ELECTROSTATIC SEPARATOR.

The great difference between the electroconductivity of graphite and that of the principal impurities associated with it in the crude concentrates early suggested that a method of separation based on

electroconductivity might be developed. Such a method has already been tried to a certain extent in the Alabama field, but the results in general have been unsatisfactory.

However, in one part of the Alabama district producing concentrates high in mica but very low in iron, the process worked well on a commercial scale and is being used there at the present time. Such material is particularly adapted for electrostatic treatment; hence the process gives satisfactory results, although certain changes in the separator as originally designed were made in order to overcome inherent mechanical defects. As this condition was only local, however, electrostatic separation was never successfully used in the rest of the district.

Improvements in the concentration mill made possible the production of concentrate carrying so much less iron that it was thought that the iron would no longer be a determining factor, and that hence the electrostatic process should offer much more chance for success than previously. Thus, in order to use this method of separation, it seemed necessary only to construct a machine that would eliminate the mechanical difficulties previously encountered in the operation of this type of apparatus.

With this end in view, a series of experiments was made with a large machine of the type at present on the market, in order to determine definitely the reason for its previous failure. However, with this machine the separation was not satisfactory. The poles of the machine are so arranged that the graphite particles receive a certain charge from one pole and are thrown into the field of the opposite pole; then the charge first obtained is neutralized and the particles are thrown in the direction of the first pole. Consequently the material progresses through the apparatus in a zigzag path, first in one direction and then in the opposite, with the result that no separation of the graphite from the nonelectroactive particles is obtained. The sand and other impurities, not being affected by the charges acting on the graphite, fall directly through the machine. Therefore, the graphite will be separated from the impurities while passing the first pole, but on passing the oppositely charged pole will be thrown in the opposite direction and into the sand from which it has just been separated. Eventually the mixture is discharged practically unchanged from its original state.

The experiments demonstrated that in order to overcome the faults mentioned it would be necessary so to design a machine that when a graphite particle had received a charge of a certain sign, it would carry this charge unneutralized, and not be forced in the opposite direction and lose its tendency to separate.

Many experiments were made with this end in view, and two types of apparatus were constructed. It was found possible to build a machine of such type that only one set of poles would tend to impart a charge to the graphite particles; these would be repelled directly into the concentrate hopper and given no chance to be neutralized and thrown back into the impurities.

Figure 19 shows the principal details of the separator as finally constructed, and indicates the manner of its operation. A view of the separator as set up ready for work is shown in Plate III.

The material to be treated is placed in the hopper at the top of the apparatus and is fed from the hopper by a roller in the bottom. The graphite first strikes one of the inclined planes that carries a weak, negative charge. While the graphite is on this plane it therefore receives also a weak, negative charge. As the plane and the graphite both have the same charge, the graphite will be repelled and will jump from the negative vane into the field induced by the positive vanes. On coming into this opposite field, the graphite gives up its weak, negative charge and receives instead a much stronger positive one, which in turn causes it to be repelled violently from the positive plate and through the openings shown between the electrodes. After having passed to the back of the plates the graphite is free to fall directly into the concentrate hopper at the bottom of the separator.

The sand and mica, being much poorer conductors of electricity, receive such a weak charge from the negative plate on which they first fall that they are not repelled into the strong, positive field, but simply slide from one negative plate to the next and so on down through the machine until finally discharged into the tailings hopper at the bottom of the apparatus and in front of the concentrates hopper.

The electrical equipment used to operate the separator consisted of a 5-k. v. a. transformer designed and built as an oil-testing transformer. It is equipped with a hand-operated voltage regulator so arranged that the voltage can be varied from zero to the maximum. While the transformer has a secondary voltage of 50,000 volts, only half of this is available for rectification as the center of the secondary coil is permanently grounded to the transformer case.

The rectifier is of the mechanical type. It consists of a rotating disk driven by a synchronous motor. Stationary shoes and revolving electrodes on the disk make and break the circuit so as to rectify alternating waves. This arrangement gives a unidirection pulsating current necessary for the operation of the machine. Figure 20 shows the wiring diagram of this equipment.

ELECTROSTATIC GRAPHITE SEPARATOR.

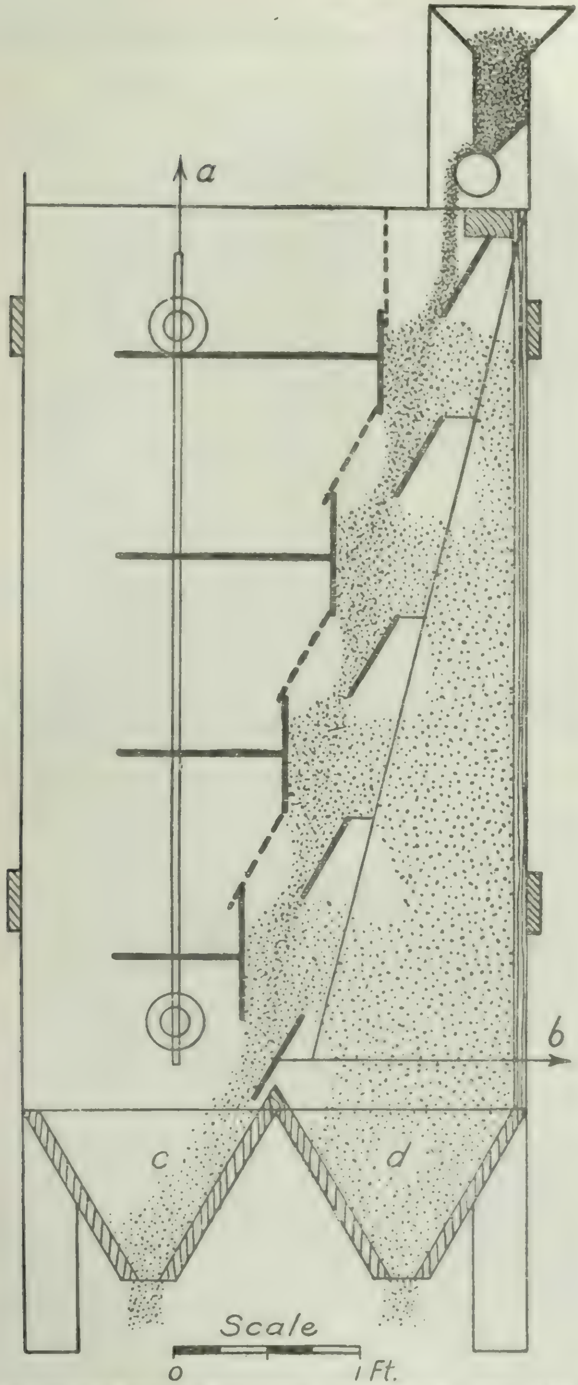


FIGURE 19.—Electrostatic graphite separator designed by Bureau of Mines. *a*, To rectifier; *b*, to ground; *c*, tailings; *d*, concentrate.

The table following gives a series of readings taken while the electrostatic separator was operated at the various voltages used in

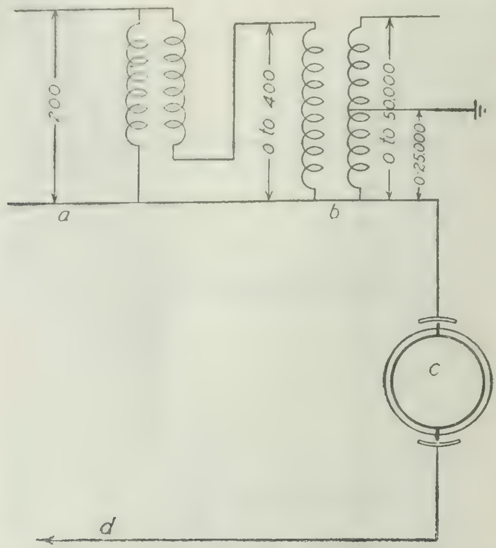


FIGURE 20.—Wiring diagram of electrical equipment used for electrostatic graphite separator designed by the Bureau of Mines.

testing. This table shows that only 200 watts is required to operate the apparatus at the highest voltage. This is equivalent to only 0.87 k. v. a.

Results of transformer readings taken in electrostatic-separator tests.

Primary coil.			Secondary coil.	
Volts.	Amperes.	Watts.	Power factor.	Volts.
446	2.22	2.12	0.21	27,900
440	1.95	2.00	.33	27,500
420	1.61	1.96	.29	26,250
388	1.15	1.52	.34	24,200
360	.90	1.32	.40	22,500
272	.50	.72	.53	17,000

The equipment (5 k. v. a.) is therefore large enough to operate a battery of six machines of the same type and size as the one described. Obviously the cost of power for the concentrating of graphite by electrostatic means will be small, an important consideration where power is costly.

After the completion of the equipment just described, a series of tests was made with the various concentrate samples in order to determine the efficiency of the device and the limitations of its use.

The voltage used in all of the tests was 28,500. The table following shows the results obtained in the treatment of the various samples.

Results of tests with electrostatic separator.

Sample No.	Percentage of graphite in—			Percentage of concentrates—	
	Feed.	Concentrates	Tailing.	Coarser than 100 mesh.	Finer than 100 mesh.
1	48.95	59.02	25.16	61.5	24.2
2	51.25	74.23	13.90	75.8	59.8
2	51.23	78.81	19.04	80.2	61.6
4	42.61	68.58	8.30	71.1	24.7
4	42.61	75.89	....	80.6	65.2
2-100	81.2	87.62	....	88.5	60.4
Buhr mill:					
1	48.95	64.00	25.00	66.2	58.4
5	73.80	83.0	52.80	63.2	73.2
4	42.60	71.20	15.50	71.6	71.0
8	42.45	67.20	17.80	77.4	62.4
9	40.44	80.40	33.00	81.6	72.4

The results obtained with the electrostatic separator can be considered most satisfactory for two reasons. The tabulated results show that in every test the grade of the concentrates was considerably higher than the material fed to the machine. This improvement was accomplished without grinding, a step that will, in spite of all precautions, cause a certain reduction in size of the original coarse material in the crude concentrates.

In each of the eleven tests, except one, the concentrates were increased in grade by 10 to 40 per cent carbon. The proportion of carbon remaining in the tailings of the electrostatic separator varied greatly, but this variation was due, in almost every instance, to the carbon that is too fine to be acted upon by the apparatus in the most efficient manner, or to the variation in the number of flakes of graphite attached to pieces of sand large enough to cause them to be drawn into the tailings. Obviously the degree of variation will depend on the condition of the concentrates themselves and not on the treatment to which they are subjected. The photomicrographs presented in connection with the discussion of flake structure on pages 66 to 67, show this condition clearly.

As the value of graphite flake for crucible making depends on the assay of the material that is coarser than 100 mesh, the value of any plan of treatment will depend on the grade of this 100-mesh material produced. For this reason, the assays of the 100-mesh part of the concentrates are given, as well as the assays of the whole product. The results of the +100-mesh assays show that out of the eleven tests reported only two of the +100-mesh concentrates contained less than 70 per cent carbon, while seven of the number contained more than 75 per cent graphite. Such results were especially

satisfactory in view of the fact that the concentrates had not been ground, with the consequent loss of coarse flake always resulting from a treatment of this nature.

It was found necessary to give the concentrates two treatments in the electrostatic separator in order to obtain optimum results. This requirement could be met in a commercial installation in one of two ways—by installing twice the number of separators required for a single treatment or by constructing apparatus containing twice the number of poles in the experimental equipment. It is probable that the latter method would prove the more satisfactory.

SUMMARY OF RESULTS WITH ELECTROSTATIC SEPARATOR.

The results obtained with the electrostatic separator prove conclusively that electrostatic separation can be successfully used in the finishing of graphite. They also indicate that the separators on the market at present will have to be changed in certain respects in order to make possible the obtaining of satisfactory results by their use. The results also seem to prove that the apparatus has a wider application than is possible with any of the other devices tested, with the possible exception of the buhr mill.

The electrostatic process seems also to be the most economical, as the cost of power is low and the loss of coarse and valuable flake is small.

At the same time, it is to be expected that, although the device will invariably improve the grade of the concentrates treated, it will not yield a finished product containing 85 per cent carbon, which is satisfactory for crucible manfacture.

However, the improvement in grade of the concentrates tested was so great as to indicate that electrostatic separation will, with few exceptions, produce a feed for the buhr mill that will be ideal for that apparatus to finish to the crucible requirements without excessive loss of coarse flake. The removal of the coarse sand was almost complete.

The results already given show that although the electrostatic separator will benefit the concentrates the degree of beneficiation will vary. Seemingly, the degree of variation depends on the physical condition of the concentrates themselves previous to treatment. In other words, the results to be obtained with this separator, like those with any other type of equipment, will depend on the concentrates themselves. Hence there can be no doubt that conditions will sometimes be met under which this type of treatment would not be as satisfactory as some other that might be developed for the removal of the coarse sand. However, the process has a more general application than any of the methods described thus far.

FLOTATION AS A FINISHING PROCESS.

Flotation as a process of concentrating the graphitic content of ores has been used for some time and with a fair degree of success. For this reason and because graphite has a much greater tendency to float than the impurities that are usually found associated with it the idea of refloating the concentrate to remove the impurities before buhr-mill treatment was suggested.

There are two primary reasons for thinking that double flotation could be used in this connection. The first is the law of concentration that the grade of the concentrates produced by a concentration process will vary directly with the richness of the feed being treated. As it is possible to produce a 45 per cent graphite concentrate from ore carrying only 3 per cent graphite, it is reasonable to believe that, under the proper conditions, it should be feasible to make a concentrate carrying, say 70 per cent or more of graphite from a feed carrying 40 per cent or more of graphite.

The other possible reason for the success of flotation has already been suggested in the discussion of the use of the pebble mill. The point in mind is the porous condition of much of the silicious material that is found in the crude flotation concentrates. It was believed that if the concentrates from the first flotation treatment were to be ground or given some other treatment by which the sand could be reduced in size sufficiently to liberate the entrained air this siliceous material could be largely removed by a second flotation treatment.

FLOTATION TESTS.

In order to prove or disprove these assumptions, three tests were made with a sample of flotation concentrate. One of these tests was made after the sample had been ground in the buhr mill, one after a treatment in the pebble mill, and one with material just as received without any treatment preliminary to the refloating.

The results obtained by this series of tests were as follows:

Results obtained in three reflotation tests.

Sample No.	Previous treatment before refloating.	Graphite in—			Recovery.
		Heads.	Concentrates.	Tails.	
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
2.......	None....................	51.23	67.35	10.8	95.7
2.......	Buhr mill...............	51.23	80.73	2.1	98.[illegible]
2.......	Pebble mill............	51.23	83.40	5.6	97.7

These results make it evident that simple reflotation, without a preliminary treatment, will materially raise the grade of the prod-

uct, and give a high recovery of the carbon content of the feed. However, as would be expected, the results do not indicate that simple reflotation would be satisfactory in commercial practice.

As regards reflotation following treatment in buhr or pebble mills, the results are such as to justify its adoption in a commercial installation.

Both the mechanical and the pneumatic types of flotation machines were used in the tests and, as far as could be noted, neither of the machines showed an advantage over the other.

Several oil mixtures were used. A mixture of 2 pounds of No. 17 oil and 0.75 pound of No. 5 oil, both made by the General Naval Stores Co., was found to be satisfactory in each test.

A series of tests was also made with sample 2 in order to determine the different results that could be obtained with different oil mixtures and various periods of agitation. These results are added for general interest, and to show what poor results can be obtained from a sample, by inefficient operation and incorrect conditions, as compared with the satisfactory results possible under proper conditions.

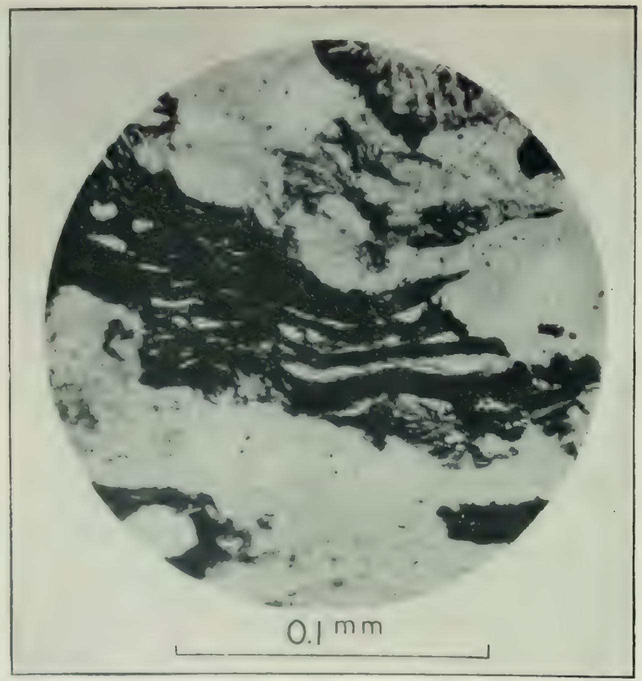
Results of refloating crude concentrates.

Sample No.	Preliminary treatment.	Percentage of graphite in—			Assay of concentrates.	
		Heads.	Concentrates.	Tails.	Coarser than 100 mesh.	Finer then 100 mesh.
2	Buhr mill	51.23	84.24	39.3	88.00	70.5
4	do	42.61	69.69	2.5	73.9	63.1
2	do	51.23	80.73	2.1	82.7	67.6
2	Pebble mill	51.23	83.10		86.0	77.1
2	None	51.23	67.35	10.8	68.4	59.3
2	Pebble mill	51.23	87.6	7.5		
2	do	51.23	76.27	17.68	78.6	49.0
2	do	51.23	83.4	5.6		

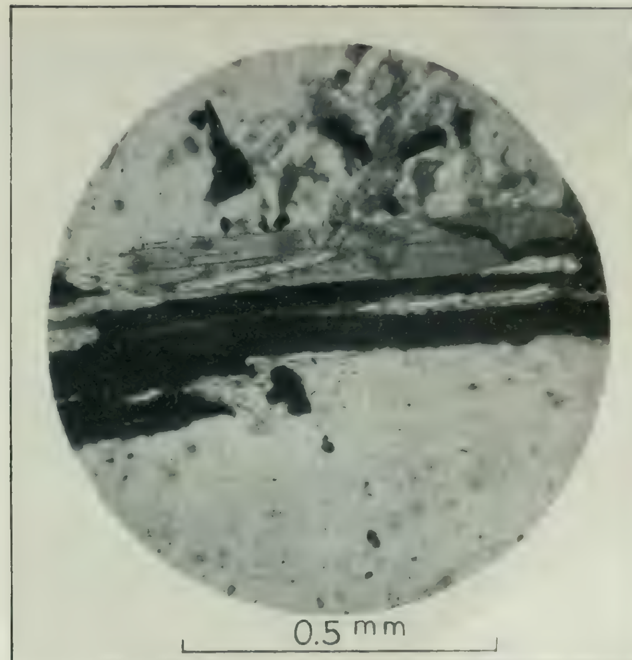
In discussing the use of flotation, it might be said that the process when used to treat concentrates that have been previously ground, for instance in a pebble mill, will produce a material that will be particularly amenable to the final treatment in a buhr mill. The results indicate that supplementary treatment will be necessary to produce material containing the required 90 per cent graphite.

THE BUHR MILL: ITS PLACE IN GRAPHITE FINISHING.

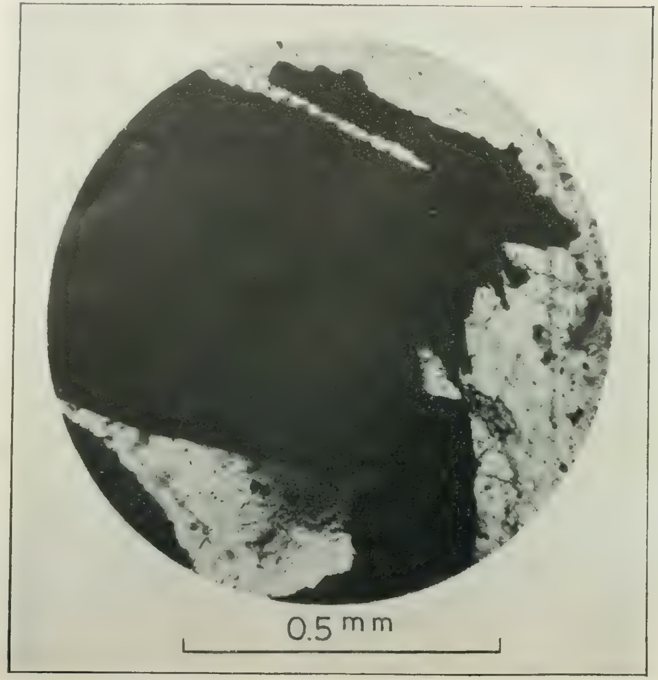
The function of the buhr mill has been previously discussed in this report. The action of the machine has also been suggested, with the two principal reasons for unsatisfactory results under certain conditions, namely, lack of uniformity in the product finished by it, and the excessive destruction of flake sometimes attending its use.



A. MICROPHOTOGRAPH OF GRAPHITE ORE, SHOWING IRREGULAR STRUCTURE.



B. MICROPHOTOGRAPH OF GRAPHITE ORE, SHOWING BANDED STRUCTURE.



A. MICROPHOTOGRAPH OF GRAPHITE ORE, SHOWING A LARGE PLATE WITH A TRIANGULAR INCLUSION.

B. MICROPHOTOGRAPH OF GRAPHITE ORE, SHOWING IMPURITIES EMBEDDED IN FLAKES.

During the comparatively long period that this machine has been used to finish graphite, methods of overcoming inherent difficulties in its use have become evident. One requirement is that the concentrate be of comparatively high grade before grinding in the buhr mill. With such material, no difficulty is encountered in consistently producing a crucible stock assaying 90 per cent graphite, provided the other conditions are correct.

In order that the excessive destruction of coarse flake incident to buhr mill grinding may be eliminated, it is necessary only to remove the coarse, gritty particles, or at least those coarse enough to require a large amount of grinding in the buhr mill.

Stated simply, when the buhr mill is used principally as a grinding machine on low-grade material, the grade of the product resulting can not be depended upon, and an excessively large proportion of the flake will be ground to dust. When, on the other hand, the machine is used only as a polishing or buffing machine, it can be trusted to give satisfactory results.

As already mentioned, the limitations of the mill were realized early in the progress of the work, and the experiments were conducted accordingly. Most of the products resulting from the various treatments contained less than the required 90 per cent of carbon and, therefore, could be used by the crucible makers only after a treatment in the buhr mill, to bring them to the necessary grade.

A view of the buhr mill used in the experimental work is shown in Plate II, *B* (p. 53).

RELATION OF FLAKE STRUCTURE TO FINISHING.

In order to show more clearly the truth of the assumptions previously stated, microscopic studies were made of graphite flakes, both before and after finishing. Also a series of microphotographs were made of this material. Four of these are reproduced in Plates IV and V.

The photographs show that the flakes were composed of series of thin laminations or plates of graphite, interbedded with thin pieces of quartz, mica, and other impurities. In some material these impurities were seen to be entirely surrounded by the carbon.

In order to determine the proportion of the impurities held in this manner, flakes of seemingly pure graphite, as it occurs naturally in the ore, were carefully picked by hand from the crude concentrate and assayed. In selecting the sample, special care was taken that nothing but the free, natural flakes were obtained for assay. The sample obtained in this manner was then carefully assayed, with the following results: Moisture, 1.26 per cent; carbon, 90.2 per cent; ash (impurities), 8.54 per cent. Examination of the ash

under the microscope showed that it consisted principally of mica with some quartz. This experiment indicated conclusively that impurities are imbedded between the laminations of the graphite, and also that the removal of all impurities not attached to the graphite flakes would give a finished product assaying only 90 per cent carbon.

This figure is, then, close to the limit of graphitic content possible in a finished material, even after treatment by a perfect finishing process.

Evidently the interlaminated impurities must be freed from the graphite flakes before they can be removed from the crude concentrate, and the only feasible method of accomplishing this is by separation of the graphite flakes into their individual laminations. It follows that the more there are of these interbedded layers to be removed, the greater will be the number of plates into which the flakes must be subdivided. Also, the greater the number of plates made in the treatment, the thinner they must be and, hence, the smaller the average diameter will tend to be.

We have seen that the value of the graphite for crucible manufacture depends largely on the diameter of the flakes composing the crucible stock. Therefore, if all of the interbedded impurities are to be removed, the resulting flakes will be so thin and of such small diameter that the value of the graphite for crucible making will be lessened.

Theoretically it would be possible so to separate the flakes into individual plates by buhr-mill grinding as to make a finished product in such a fine state of division that it will be valueless. Hence, the degree to which the finishing can be accomplished in a commercial plant will depend on the number and the characteristics of the interbedded impurities in the crude flakes. These factors will also determine the amount of grinding that is permissible in the buhr mill to permit removal of impurities, and will determine the grade of finished product that will be produced by a definite amount of such grinding. The proportion of dust produced will also depend on the same factors.

In other words, the interbedded material is the important factor in determining the economic possibilities of the finishing process. If the flake graphite contained in a certain crude concentrate is of such a nature that, in order to produce 90 per cent carbon in the finished product, an undue proportion of the carbon coarser than 100 mesh is destroyed, obviously the product will not be satisfactory.

There is no doubt that the buhr mill is the best commercial apparatus that has been developed to date for separating the flakes of graphite into their individual laminations and at the same time re-

ducing the freed impurities to a size permitting their removal by screening or bolting.

Several important features must be considered in grinding graphite concentrates in a buhr mill. The first point to be emphasized is that in order to produce a good crucible stock it is necessary to do more than remove the gangue present in the crude concentrates; it is also imperative that impurities interfoliated between the graphite laminations be removed. Clearly, in order to remove such impurities, some sort of grinding or rubbing process must be used, and, preferably, this should follow the removal of superficial or incidental impurities, so that these can not act as abrasive on the graphite flakes during the grinding.

From a survey of the grinding or polishing apparatus available, the conclusion is reached that the buhr mill is peculiarly adapted for this work, and that it is the only machine that can be depended on to do the required work in a satisfactory manner.

The results of the experiments indicate that the efficiency of the buhr mill, and hence of the whole finishing process, will depend on the character of the material that is being ground, and this in turn will depend both on the character of the flakes as they occur in the ore and on their treatment previous to grinding in the buhr mill. No hard and fast rules can be prescribed for the treatment of any particular concentrate, but in order to determine the most efficient treatment, it will be necessary to ascertain by experiment the best system to be followed.

The discussion indicates that satisfactory results depend on so many and such varied factors that undoubtedly there are certain concentrates produced from particular ore that can not be made into a crucible stock at a profit. Hence, it is true that although the equipment, the process, and the skill used in operation are all important for successful results, there is the added necessity that the flake itself meet certain requirements.

IMPURITIES IN GRAPHITE.

In order to learn more of the chemical nature of the impurities that were encountered in the crude concentrates, and in the finished graphitic material, chemical analyses were made of ash samples from some of the representative graphite samples. The results are presented in the table following.

Results of analyses of samples of graphite ash.

Constituent.	Ash from graphite sample 2.	Ash from graphite sample 4.	Ash from sample 2 after special treatment.[a]	Ash from sample 2 after special treatment.[b]
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
SiO_2	65.80	78.71	45.46	51.80
Al_2O_3	20.53	11.45	36.90	33.75
Fe_2O_3	10.77	8.65	14.00	11.85
CaO	.80	.73	.75	.67
Na_2O and K_2O	2.46	.71	2.68	2.54
Total	100.36	100.25	99.79	99.91

[a] Treated in electrostatic separator and in buhr mill.
[b] Treated in buhr mill, given flotation treatment, and again put through buhr mill.

These results indicate that the finishing process tends to remove the silica but causes the alumina to be concentrated. This is, of course, due to the removal of the silica in a greater proportion than the mica, which contains the greater part of the aluminum oxide. This result is to be expected for the reason that the silica can be more easily ground because of its more massive form and brittle character.

It should be mentioned that mica, because of its flat shape and its toughness, is probably the most difficult impurity to remove in graphite refining.

RESULTS FROM COMBINATION OF VARIOUS TREATMENTS.

After considering the various apparatus that might be used in the production of crucible stock from crude concentrates, and a few of the factors that might be expected to affect the results, experiments were made to determine the results that could be obtained by logical combinations of the various schemes and apparatus.

The results already obtained indicated that two general schemes could be developed for a complete finishing process to produce crucible stock from Alabama concentrates. One of these schemes may be termed a wet process and the other a dry process.

The dry process is rather simple and involves only a treatment of the crude concentrate in the electrostatic separator, followed by grinding in the buhr mill and screening. The coarse sizes from the screen constitute the finished graphite suitable for crucible manufacture and these were found to carry 90 per cent carbon and flakes of the correct size for crucible manufacture.

The wet process is also simple, though involving one more step than the dry process. Wet concentrates are used in each step of this process, except that in the last step, grinding in the buhr mill, dry concentrates are used. Thus, the wet process should offer some advantages over the dry process. The wet concentrates are ground in the pebble mill for a period of time previously determined to be correct. The ground material is then floated in a standard flotation machine. The concentrates produced are dried and reground in the

buhr mill, after which they are screened. The screen used is 100 mesh and the oversize is the finished crucible stock.

A test of the dry process was made with sample 2, which assayed 51.23 per cent carbon. The material was twice treated in the electrostatic separator, a small sample was cut from the concentrates for analysis, and the remainder ground in the buhr mill. The product from this grinding was then screened and assayed. The whole product from the electrostatic treatment assayed 74.23 per cent carbon; the +100-mesh material contained 75.8 per cent and the −100-mesh substance, 59.8 per cent. The whole product from the grinding in the buhr mill assayed 78 per cent carbon; the −100-mesh material showed 45 per cent carbon, and the +100-mesh product, or crucible stock, 90.5 per cent. The total recovery of graphite in the finished crucible stock was 83 per cent of the graphite in the original sample.

The product obtained by grinding the concentrate from the electrostatic separator in the buhr mill was submitted to screen analysis and assay with the following results:

Results of use of electrostatic separator and of buhr mill.

Mesh.	Percentage of each size, by weight.	Carbon assay. (per cent).	Units of carbon, each size.[a]	Percentage of total carbon.[b]
+14	Tr.			
+20	Tr.			
28	0.9	92.8	83.00	1.06
35	5.1	91.8	468.00	6.00
48	15.2	91.4	1,388.00	17.80
65	23.0	90.1	2,054.00	26.30
100	26.3	86.2	2,265.00	29.00
150	13.8	85.6	1,181.00	15.10
200	4.3	39.8	171.00	2.20
−200	10.8	17.4	188.00	2.41
Total	99.4	78.4	7,798.00	99.87

a Product of column 2 times column 3.
b Results obtained by dividing figures in column 4 by sum total of column 4.

The following tabulation shows calculated results based on the foregoing data and also the results of actual analyses:

Selected data showing results of use of electrostatic separator and of buhr mill.

Material.	Percentage of final product.	Carbon assay (per cent).	Units of carbon.[a]	Percentage of total carbon.[b]
Calculated results:				
Crucible stock (material over 100 mesh)	70.5	88.8	6,258.6	80.2
Dust (material less than 100 mesh)	28.9	53.3	1,540.0	19.8
Total	99.4	78.4	7,798.6	100.0
Results of analyses:				
Crucible stock	70.5	90.5	6,380.2	83.1
Dust	28.9	45.0	1,300.5	16.9
Total	99.4	77.2	7,680.7	100.0

a Column 2 times column 3.
b Results obtained by dividing figures in column 4 by sum total of column 4.

These results were exceptionally satisfactory. The grade of the coarse material was higher than the 90 per cent product required by the crucible makers, whereas the loss of less than 20 per cent of the total graphite in the original sample was lower than the loss in the finishing mills that are producing graphite of crucible grade from the Alabama concentrates.

As already mentioned, the whole success of a finishing process depends on the removal of a large percentage of coarse hard impurities that tend to cause excessive destruction of valuable flake in the buhr mill grinding. Such removal is, of course, the function of the electrostatic treatment, and the results prove how satisfactory such treatment was.

A test of the wet method was next made with 400 grams of sample 2 concentrate. The material was ground with 600 c. c. of water for one hour and floated in the Callow pneumatic machine. The concentrate from this treatment, when filtered and dried, contained 83 per cent carbon. This concentrate was put through the buhr mill, when the +100-mesh material was found to contain 90.3 per cent carbon. The recovery of the graphite was 80.2 per cent of the carbon in the original sample. Tabulated results follow.

Results of treating concentrates in pebble mill, flotation machine, and buhr mill.

Mesh.	Percentage, by weight, of each size.	Carbon assay (per cent).	Units of carbon, each size.[a]	Percentage of total carbon.[b]
14	None.			
20				
28	1.2	88.6	106.00	1.29
35	5.9	94.0	554.00	6.70
48	16.0	90.8	1,455.00	17.61
65	24.8	93.0	2,305.00	27.94
100	26.6	89.0	2,365.00	28.65
150	12.9	83.4	1,076.00	13.01
200	13.5	63.6	223.00	2.70
−200	10.1	17.4	167.00	2.02
Total	100.0	82.5	8,251.00	99.91

[a] Column 2 by column 3.
[b] Results obtained by dividing figures in column 4 by sum total of column 4.

The following tabulation shows results based on the foregoing data:

Selected data showing results of use of pebble mill, flotation machine, and buhr mill.

Material.	Percentage of final products.	Carbon assay (per cent).	Units of carbon, each size.[a]	Percentage of original carbon.[b]
Crucible stock (material over 100 mesh)	73.5	91.0	6,690.0	81.5
Dust (material less than 100 mesh)	26.5	57.5	1,525.0	18.5
Total	100.0	82.5	8,215.0	100.0

[a] Column 2 times column 3.
[b] Results obtained by dividing figures in column 4 by sum total of column 4.

As with the dry process, the success of this treatment depends on the removal of the coarse impurities before the final buhr mill grinding, and such removal is accomplished by the grinding in the pebble mill followed by flotation.

Owing to the short time available for the graphite investigation it was impossible to test all the samples with the combination treatment, but there can be no doubt that the results of such treatment would check closely with the results already described.

GENERAL SUMMARY AND DISCUSSION OF RESULTS.

As a result of the investigations made to determine the most satisfactory methods of refining crude graphite concentrates, several important facts were developed.

Probably the most important of these is that each concentrate to be treated is a problem in itself, hence no treatment applicable to all kinds of concentrate can be prescribed. Such a limitation is due to the differences between the characteristics of the crude graphite flakes and those of their associated impurities. The most important characteristics that will affect the type of treatment to be applied are the hardness, toughness, diameter, and thickness of the flakes and their contents of interfoliated impurities. The different physical properties affect the amount and intensity of the grinding necessary to remove the impurities without the destruction of too large a proportional amount of the large and more valuable flakes.

Another important factor in determining a satisfactory finishing process is the kind of impurities that must be removed. When these impurities exist free from the flakes of carbon they can be removed, to a great degree, with a small loss of coarse carbon. When these impurities are soft and of small size they can be removed after a slight and nondestructive grinding operation, which will in all probability cause a comparatively small loss of valuable flake.

On the other hand, coarse, hard impurities are much more difficult to handle in a satisfactory manner. This type of material offers the greatest problem to the operator of the finishing mill. The results obtained in the experiments indicate that the success of the finishing process will depend on the success obtained in the removal of this sort of impurities at the earlier stages of the finishing operations.

The investigations have also brought out the fact that the buhr mill is a necessary apparatus in all processes for graphite finishing. The buhr mill separates the flakes into their constituent laminations and prepares them for the removal of the interbedded or interfoliated impurities.

The aspirator and the pneumatic jig were found to have an application under certain circumstances. It was seen, however, that such apparatus can be considered only for preliminary treatment previous to the regular finishing process and should be used only for the removal of very coarse, hard, free impurities that are encountered from time to time.

The experiments proved fairly conclusively that the use of either the electrostatic separator or the pebble mill, followed by flotation, will be applicable to all types of concentrates produced in the Alabama fields. The material produced by either of these types of apparatus can be ground in the buhr mill with satisfactory results.

The different processes of finishing graphite may be classified under two general heads—wet processes and dry processes. The decision as to which of the two should be used is dependent on the characteristics of the concentrates themselves. It is important that neither be adopted except after the necessary experiments and on the advice of an engineer thoroughly conversant with the problems to be encountered in the treatment of the particular concentrate in mind and with the economic conditions that will exist in the plant.

It can be said that with rare exceptions a satisfactory crucible stock can be produced from all of the concentrates that are at present made from the Alabama ores, and that the recovery of +100-mesh flake at the majority of finishing plants now operating can probably be improved. It should be possible to keep the recovery of material coarser than 100 mesh at 70 per cent of the total carbon in the original concentrates, and in many instances this can be increased to 80 per cent or more. These figures are sufficiently high to make profitable many of the plants that are not now operated except at a loss.

In conclusion, it should be emphasized that the results obtained in finishing the crude graphite concentrates are dependent largely on the character of the crude flake itself as contained in the ores. Moreover, mills that are producing a concentrate containing large tough flake in their concentrating plants, can also produce a satisfactory crucible stock at a profit from a finishing plant which has been correctly designed and intelligently managed. The operator who is unfortunate in having to mine small, low-grade flake requiring a large amount of intensive grinding, and which is associated with a large quantity of coarse siliceous or micaceous impurities, is bound to have difficulty in producing the required grade of crucible stock at a profit.

As in all mining ventures, success or failure depends more on the character of the raw ore than on the treatment given the ore after it has been mined.

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INDEX.

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